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DEPARTMENT OF DEFENCE

DEFENCE SCIENCE AND TECHNOLOGY ORGANISATION

MATERIALS RESEARCH LABORATORIES

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REPORT

MRL-R-1043

A COMPARISON BETWEEN SEVERAL STANDARD METHODS USED TO CHARACTERIZE THE IGNITION/IGNITION TRANSFER OF PYROTECHNIC COMPOSITIONS - A COLLABORATIVE STUDY.

PART I : DATA

P. Barnes, L. de Yong, J. Domanico, P. Twadawa, and F. Valenta

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ABSTRACT

A collaborative study has been made of samples of pyrotechnic compositions (delays, coloured smokes and a percussion primer), by a number of laboratories. A variety of techniques have been used to characterize the ignition/ignition transfer behaviour of the compositions. These include temperature of ignition, heat of reaction, burning rate, time to ignition, safety tests and DSC/DTA analysis. This report presents the data collected by each laboratory during this investigation.

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ABSTRACT

A collaborative study has been made of samples of pyrotechnic compositions (delays, coloured smokes and a percussion primer) by a number of laboratories. A variety of techniques have been used to characterize the ignition/ignition transfer behaviour of the compositions. These include temperature of ignition, heat of reaction, burning rate, time to ignition, safety tests and DSC/DTA analysis. This report presents the data collected by each laboratory during this investigation.

C O N T E N T S

CONTENTS
(Continued)

A COMPARISON BETWEEN SEVERAL STANDARD METHODS USED TO CHARACTERIZE THE IGNITION/IGNITION TRANSFER OF PYROTECHNIC COMPOSITIONS - A COLLABORATIVE STUDY

1. INTRODUCTION TO MATERIALS EXCHANGE PROGRAMME

Malfunction of a pyrotechnic device or the pyrotechnic component of a device can cause failure of a complete weapon system which has operational consequences and wastes time and money. One of the major causes of these types of malfunctions is the energy or ignition transfer process between the various components being interrupted or proceeding in an unexpected manner.

The philosophy currently reflected in efforts directed at solving these pyrotechnic ignition/ignition transfer problems, focusses on either the materials in the pyrotechnic composition (system independent approach) or on specific in-service problems associated with a specific application (system dependent approach). These approaches generally lead to the problem being solved in the shortest possible time to satisfy a specification rather than relying on fundamental research which could lead to a more complete understanding of the problem as well as eliminating the cause.

Many techniques have been developed in an attempt to measure or characterize the ignition/ignition transfer behaviour of pyrotechnics. However, it is often unclear which test is relevant to the problem at hand and whether the method is adequate in predicting variations in a filled device.

To examine some of the major methods that are being used, a programme of work was initiated in which five laboratories exchanged a range of pyrotechnic compositions and subjected them to several standard tests. This report presents only the data obtained during the course of this investigation and details the test methods used. Interpretation and analysis of the data will be the subject of a future report.

1.1 Choice of Materials

A range of pyrotechnic compositions comprising coloured smokes, delays and percussion primers was chosen for examination. They were chosen to represent the most commonly used pyrotechnics in munitions. Each of the materials was supplied either as a loose composition or as a pressed filling in a filled store which had to be broken down and the composition extracted. Details of the compositions are given in Appendix 1.

1.2 Laboratories Involved

The following laboratories made inputs:

Australia - Materials Research Laboratories (MRL)
Canada - Defence Research Establishment Valcartier (DREV)
UK - Royal Armament Research and Development Establishment (RARDE)
US - Naval Ordnance Station (NOS)
Chemical Research Development and Engineering Center (CRDEC)

1.3 Tests Conducted

Each composition was subjected to a series of tests which fall into the following categories:

1. Thermal characterisation (Differential Scanning Calorimetry (DSC), Differential Thermal Analysis (DTA), temperature of ignition etc.)
2. Performance (gap test, time to ignition)
3. Safety (impact, electrostatic, thermal)

A list of the specific tests conducted by each laboratory is given in Appendix 2.

The testing programme was broken into two sections - Phase 1 involved the work described above whilst Phase 2 involved Differential Scanning Calorimetry (DSC)/Differential Thermal Analysis (DTA) and burning rate determinations on one of the UK gasless delay compositions, SR37, using closely defined conditions (DSC using controlled sample weights (0-25 mg) and heating rates (5 or 10 K min^{-1})).

2. RESULTS OF AUSTRALIAN TESTING

2.1 UK and US Delay Compositions

2.1.1 Thermal Characterisation

(a) Differential Scanning Calorimetry (DSC):

DSC analysis was carried out on all pyrotechnic compositions using a Perkin Elmer DSC operating in the non-isothermal mode controlled by a Perkin Elmer Model 3600 Data Station with appropriate DSC software. All samples were accurately weighed (1-10 mg) on a Mettler ME30 analytical balance directly into aluminium or copper sample pans and the lids placed over the samples. The copper pans were used for SR37 and SR38 to achieve the high temperatures required (800 K - 950 K). The following operating conditions were used for all experiments:-

Purge gas:	Nitrogen (20-25 ml min^{-1})
Temperature calibration:	indium (429.8 K), tin (505 K), lead (600.6 K), potassium sulphate (858 K).
Heat of reaction standard:	indium (28.5 J g^{-1})
Temperature range scanned:	ambient to ca. 950 K
Scan Rate:	5 K min^{-1} or 10 K min^{-1}

Typical DSC traces for all the UK and US delays are shown in Figures 1 and 2. Analysis in terms of the heat of reaction and positions of important exotherms and endotherms is given in Table 1. These results have been corrected using potassium sulphate or indium standards as required.

TABLE 1

DSC Results for UK and US Delays[1]

Composition	Mass of Sample (mg)	Peak 1 T_{onset} (K)	Peak 1 T_{max} (K)	Peak 1 ΔH (J g^{-1})	Peak 2 T_{onset} (K)	Peak 2 T_{max} (K)	Peak 2 ΔH (J g^{-1})
IHM-BD-10-85	5.00,5.12	N	N	N			
IHM-BD-11-85	4.99,5.02	N	N	N			
IHM-TV-2-85	5.38	577.0	580.3	+ 6.7	760.7	792.5	- 279.0
IHM-WD-3-85	2.00	643.6	660.9	- 98.3	729.6	732.5	- 53.1
IHM-WD-9-85	4.94	575.2	577.9	+ 11.3	761.8	779.4	- 702.9
SR37	3.65	788.5	803.7	-1387.4[2]	840.0	868.0	-
SR38	3.49,3.65	801.1	811.8	-1464.0	-	-	-

1 Heating rate 5 K min^{-1}.
2 Mixture of 2 peaks, shoulder ca 816.4 K.
N Denotes no observable reaction.

2.1.2 Performance Testing

(a) Standoff Test (Ignitability):

Tests were conducted with the MRL Standoff Test [1] to measure the relative ignitability of each of the delay compositions. Two types of ignition stimuli were used - the standard gassy M42 percussion primer and the MRL developed gasless M42F1 percussion primer. These tests provided a comparison between the ignition effectiveness of these two primers. A more detailed explanation of the test is given in Appendix 3 and Reference [1]. Appendix 4 gives the chemical composition of the two percussion primers used.

The Standoff Test gives the physical gap, in millimetres, over which ignition is transferred between the percussion primer (the donor) and the pyrotechnic delay (the acceptor) for 50% probability of ignition of the pyrotechnic delay. The results are given in Table 2. Samples that showed no ignitions at the normal pressing load of 8.25 kN were subsequently retested at lower pressing loads down to 1.25 kN.

TABLE 2

Standoff Distances for UK and US Delays with M42 and M42F1 Percussion Primers

Acceptor Composition	Donor			
	M42 Primer		M42F1 Primer	
	$\overline{SD}$ (mm)	σ_R	$\overline{SD}$ (mm)	σ_R
SR37	78.6	16.6	301.7	30.4
SR38	49.0	4.0	46.0	14.0
IHM-BD-10-85	61.0	5.2	26.4	8.9
IHM-BD-11-85	84.6	6.3	386.3	124.0
IHM-WD-3-85	51.4	10.7	36.4	25.2
IHM-WD-9-85	30.5	13.6	27.5	7.4
IHM-TV-2-85	57.5	6.7	33.1	22.8

2.1.3 Safety Testing

The sensitivity of each pyrotechnic composition to heat, impact and electrostatic discharge was determined using the RARDE Temperature of Ignition Test (T of I), Figure of Insensitiveness Test (F of I) and spark sensitivity tests respectively. A detailed description of these tests is given in Appendix 5 and the results are given in Table 3.

TABLE 3

Sensitivity Data for UK and US Delays

Composition	F of I[1]	T of I (K)	Spark Sensitivity (J)
SR37	110	>673	<0.045
SR38	>200	>673	<0.45
IHM-BD-10-85	>200	>673	>4.5
IHM-BD-11-85	>200	>673	<0.045
IHM-WD-3-85	>200	>673	<0.45
IHM-WD-9-85	>200	>673	>4.5
IHM-TV-2-85	>200	>673	>4.5

1 Relative to RDX (F of I = 80).

2.2 Canadian, UK and US Smoke Compositions

MRL received 4 of each of the US Coloured Smoke Grenades (M-18 series) and 4 of each of the UK Coloured Smoke Grenades (L46, L47, L48 and L49). These were broken down, the composition extracted and stored in a sealed jar. No samples of the Canadian smoke composition were received.

2.2.1 Thermal Characterisation

(a) Differential Scanning Calorimetry (DSC):

The procedure was identical to that used for the delay compositions (see Section 2.1.1 (a)). Typical DSC traces for the smoke compositions are shown in Figures 3 and 4. Analysis in terms of the position of the major peaks (endotherms and exotherms) and the heats of reaction at two heating rates is given in Table 4.

2.2.2 Performance Testing

(a) Standoff Test (Ignitability):

The procedure was identical to that used for the delay compositions (see Section 2.1.2(a)) and the results are presented in Table 5. The minimum distance between donor and acceptor is 20 mm.

TABLE 4

DSC Results for UK and US Smoke Composition at Different Heating Rates

Compositions	Sample Mass(mg)	Peak 1			Peak 2			Peak 3		
		T_{onset} (K)	T_{max} (K)	ΔH (J g^{-1})	T_{onset} (K)	T_{max} (K)	ΔH (J g^{-1})	T_{onset} (K)	T_{max} (K)	ΔH (J g^{-1})
US Coloured Smokes										
1. Heating Rate 5 K min^{-1}										
M-18 Yellow	2.93,2.81	450.9	458.4	+ 148.9	478.7	483.9	-679.9	612.3	613.3	-47.3
M-18 Red	2.95,3.08	445.9	455.1	+192.0	499.2	507.9	-102.5	574.9	578.5	-282.8
M-18 Green	3.03,2.92	465.3	467.6	-332.6	576.8	615.3	-269.9	Complex set of 3 peaks		
M-18 Violet	2.92,2.86	506.3	514.6	-156.9	594.3	609.4	-794.5	759.2	794.6	-712.1
2. Heating Rate 10 K min^{-1}										
M-18 Yellow	2.80,2.87	457.4	465.6	+197.1	489.3	495.5	-673.2	612.1	614.3	-15.9
M-18 Red	2.85,3.09	425.1	432.4	+22.2	454.1	463.4	+198.7	508.9	519.1	-924.2
M-18 Green	2.89,3.03	472.2	474.3	-350.6	604.3	613.2	-142.2	642.2	650.1	-119.2
M-18 Violet	2.86,3.16	455.0	476.4	+268.6	515.6	521.6	-230.1	596.0	609.3	-655.2
UK Coloured Smokes										
1. Heating Rate 5 K min^{-1}										
PN758M	3.20,3.15	455.6	462.9	-197.1	603.6	611.3	-822.6			
PN759	3.28,3.11	461.4	468.8	-184.9	606.3	612.9	-655.6			
PN757	3.22,3.25	457.4	471.9	-660.2	609.6	612.9	-633.9			
PN760	3.11,3.01	453.9	462.6	-262.3	607.5	613.2	-873.6			
2. Heating Rate 10 K min^{-1}										
PN758M	2.95,3.14	462.8	468.0	-597.5	599.4	610.6	-745.2			
PN759	2.95,2.99	455.7	460.6	-548.1	606.4	609.5	-370.1			
PN757	3.01,2.89	462.7	478.6	-819.2	611.1	614.4	-344.8			
PN760	2.90,2.90	459.3	470.9	-320.1	609.9	612.2	-613.8			

TABLE 5

Standoff Distances for UK and US Smoke Compositions [1]

Acceptor Composition	Donor	
	M42 Primer	M42F1 Primer
	$\overline{SD}$ (mm)	$\overline{SD}$ (mm)
PN758 M	N	N
PN759	N	N
PN757	N	N
PN760	N	N
M-18 Green	N	N
M-18 Yellow	N	N
M-18 Red	N	N
M-18 Violet	N	N

N Denotes no ignition at 20 mm.
1 Pressing load 1.25 kN

2.2.3 Safety Testing

The sensitiveness of each composition to heat and impact was determined using the RARDE T of I and F of I tests. A detailed description of the tests is given in Appendix 5 and the results are given in Table 6.

TABLE 6

Sensitiveness Data for UK and US Smoke Compositions

Composition	F of I[1]	T of I (K)
PN758M	>200	463
PN759	>200	473
PN757	>200	471
PN760	>200	467
M-18 Green	130	470
M-18 Yellow	150	673
M-18 Red	120	456
M-18 Violet	180	473

1 Relative to RDX (F of I = 80)

2.3 Australian M42F1 Percussion Primers

2.3.1 Ignition Effectiveness

The ignition effectiveness of the M42F1 primer compared to the standard M42 primer (see Appendix 4 for details of primer compositions) was examined using the same procedure as outlined in Section 2.1.2 (a) and Appendix 3. Typical results are shown in Table 7 (ex Reference [1]).

TABLE 7

Values of Standoff Distance for 50% Probability of Ignition of Pyrotechnics with M42 and M42F1 Primers

Acceptor Composition	M42 Primer			M42F1 Primer		
	$\overline{SD}$ (mm)	$\frac{\sigma}{SD}$	d/σ_R	$\overline{SD}$(mm)	$\frac{\sigma}{SD}$	d/σ_R
B/Fe_2O_3(20:80)	60.7	3.3	0.5	348.0	7.2	1.3
B/PbO(10:90)	166.7	4.2	1.5	333.3	12.3	1.2
B/Pb_3O_4(10:90)	172.5	5.8	1.6	331.9	13.1	0.6
SR92	95.7	12.2	0.2	293.0	2.9	4.0
SR44	63.3	2.7	2.5	260.4	7.2	1.0
B/CuO(15:85)	78.1	6.4	0.8	254.6	8.7	0.6
B/Fe_2O_3(25:75)	183.0	6.9	1.1	252.6	7.3	1.0
SR112	64.3	1.8	1.7	252.9	13.1	0.5
G20	71.7	2.4	1.2	225.8	5.8	1.5
SR252	70.0	4.4	0.6	67.3	3.7	0.7
B/Fe_3O_4(15:85)	53.2	1.3	2.0	21.3	1.4	1.1

$\overline{SD}$ is the mean standoff distance

$\frac{\sigma}{SD}$ is the standard deviation of $\overline{SD}$

σ_R is the population standard deviation and

d is the testing increment height.

2.3.2 Pressure Testing

Comparative tests were conducted to measure the amount of gas evolved by the M42F1 primer and the M42 primer. The primers were fired into a fixed volume of 0.5 cm^3 and the pressure/time behaviour was measured using a Kistler pressure transducer. Traces for both primers are shown in Figure 5 and the average peak pressure of 6 results is given in Table 8 below:

TABLE 8

Average Peak Gas Pressure for M42F1 and M42 Primers

	M42F1 Primer	M42 Primer
Pressure (MPa)	2.8	35.0

2.4 Phase 2: DSC/DTA on UK Delay SR37

The effect of changes in sample weight and heating rate on the DSC analysis of SR37 was studied in Phase 2. Duplicate results are shown in Table 9.

3. RESULTS OF CANADIAN TESTING

3.1 UK and US Delay Compositions

3.1.1 Thermal Characterization

(a) Differential Thermal Analysis (DTA) and Thermogravimetric Analysis (TGA):

DTA and TGA studies were carried out simultaneously on a Mettler TA-2 thermoanalyser operated in the temperature range of ambient to 1073 K. All runs were carried out in platinum crucibles using an atmosphere of flowing helium (50 ml min^{-1}). Aluminium oxide was used as the reference material. All samples were accurately weighed on a Mettler ME 22 Analytical balance directly into the crucibles. The results for selected compositions are shown in Table 10 and typical DTA traces are shown in Figure 6. Thermogravimetric data is also shown in Table 10.

TABLE 9

DSC Exotherms for Delay Composition SR37

Sample Mass (mg)	Heating Rate (K min^{-1})	Exotherm 1[1]			Exotherm 2[1]		
		T_{onset} (K)	T_{max} (K)	ΔH (J g^{-1})	T_{onset} (K)	T_{max} (K)	ΔH (J g^{-1})
3.56	5.0	788.5	803.7	1387.4	-	-	-
3.61/3.62	10.0	804.9/804.5	819.8/820.7	453.9/421.7	858.4	879.6	36.2
10.00/9.99	5.0	794.6/792.5	808.0/806.3	223.8/154.8	852.4/847.1	869.7/870.1	58.2/74.5
10.06/10.08	10.0	808.1/804.7	821.1/819.5	632.2/161.1	857.9/853.1	877.7/880.7	20.9/80.8

1 Duplicate results.

TABLE 10

DTA Characteristics of Selected UK and US Delay Compositions Heated at 10 K min^{-1}

Composition	Sample Mass (mg)	Temperatures (K) Exotherm 1		Exotherm 2		Exotherm 3		Exotherm 4		Residue (mg)
		T_{onset}	T_{max}	T_{onset}	T_{max}	T_{onset}	T_{max}	T_{onset}	T_{max}	
SR37	5.10	759	805	824	879	–	–	–	–	4.36
SR37[1]	5.00	732	800	822	889	–	–	–	–	–
IHM-WD-3-85	5.03	677	695	743	774	790	797	932	964	5.33
IHM-BD-11-85	4.93	981	1049	–	–	–	–	–	–	5.7
IHM-BD-11-85	5.02	985	1051	–	–	–	–	–	–	–
IHM-TV-2-85	5.11	748	779	835	891	–	–	–	–	5.22
IHM-TV-2-85	5.02	749	775	840	894	–	–	–	–	5.11

1 Heating rate 5 K min^{-1}.

(b) Combustion Calorimetry (Heat of Reaction):

The heat of reaction of powdered samples was determined using a Parr calorimeter bomb under 1 atmosphere, initial pressure, of argon. An ignition composition (50% Si, 20% PbO_2 and 30% CuO) was used as the normal hot wire system could not achieve ignition and the heat output of the ignition composition was subtracted from the total heat output. The results are shown in Table 11.

TABLE 11

Thermal, Performance and Safety Data for UK and US Delay Compositions

Composition	Temperature of Ignition (K)	Heat of Reaction[1] ($J\ g^{-1}$)	Density[2] ($g\ cm^{-3}$)	Average Burning Rate[3] ($mm\ s^{-1}$)
SR-37	>623	2401.6	2.46	21.2
SR-38	>623	1723.8	2.51	9.3
IHM-BD-10-85	>623	1631.8	2.64	12.3
IHM-BD-11-85	>623	2188.2	2.37	40.3
IHM-WD-3-85	>623	736.4	4.59	6.8
IHM-WD-9-85	>623	1389.1	3.51	2.3
IHM-TV-2-85	>623	1142.2	4.64	3.6

1 Average of three measurements.
2 Average of two samples.
3 Average for two sets of tests at ambient temperature.

3.1.2 Performance Testing

(a) Burning Rate:

The average propagation rates were measured using a thermocouple system. Three chromel-alumel thermocouples were used per test, with the results recorded on a Honeywell Visicorder. Powdered smoke compositions were loaded into a phenolic impregnated cardboard tube with an inside diameter of 13.2 mm while delay compositions were loaded in a 6.5 mm inside diameter copper tube. All samples were loaded in a series of ten pressings at a pressure of 154 MPa with a hydraulic press, keeping the total length of the pressed column between 50.8 mm and 57.2 mm. In the case of the delay compositions, a 0.2 g increment of ignition composition SR-252 [2] was pressed on top of the column to ensure a good ignition. Strands of the Canadian castable smoke compositions, 12.7 mm square by 50.8 mm long, were simply cut out from the appropriate blocks and their surface inhibited with an epoxy coating. The sample densities were estimated from sample masses and volumes. The thermocouples, spaced 12.7 mm apart, were inserted into holes

drilled halfway through the column. Figure 8 shows the geometry of the prepared samples. All samples were burned at ambient temperature, in vertical position and ignited at the top end using either the open flame of a torch (smoke compositions) or an electric squib (delay compositions).

Table 11 gives the results for both the US and the UK delays and Figure 7 gives a typical recording showing the response of the thermocouples.

3.1.3 Safety Testing

(a) Temperature of Ignition:

The temperature of ignition was determined by the Canadian Explosives Research Laboratory (CANMET) of Energy, Mines and Resources Canada using the Wood's-metal bath technique. The results are given in Table 11.

3.2 Canadian, UK and US Smokes

3.2.1 Thermal Characterisation

(a) Differential Thermal Analysis (DTA) and Thermal Gravimetric Analysis (TGA):

The procedure used here was identical to that used for the delay compositions (see section 3.1.1(a)). Typical DTA/TGA traces for the US, UK and Canadian Red smoke compositions are illustrated in Figure 9. Values of the peak temperatures are given in Table 12.

TABLE 12

DTA Peak Temperatures for Canadian, UK and US Red Smoke Compositions

Sample	Sample Mass (mg)	Peak Temperatures (K)	
		Endotherms	Exotherms
SK-338	4.0	438, 462	490, 586, 718, 785, 905
SK-338	2.06	439, 459	492, 593, 715, 786
M-18 Red	4.86	389, 433, 462	513, 576, 600, 786
M-18 Red	5.08	390, 434, 465	518, 577[1]
PN757	5.07	419, 443	479, 605, 671[1]

1 Test stopped at T < 700 K

(b) Heat of Reaction:

The heat of reaction was determined using a Parr Bomb Calorimeter (see section 3.1.1 (b)). The US and UK powdered smoke compositions were pressed into pellets and the Canadian castable smoke compositions (SK series) were used as provided - samples were simply cut out from the blocks supplied. Table 13 gives the results for all the smoke compositions.

TABLE 13

Summary of Results for Canadian, UK and US Smoke Compositions

Composition	Temperature of Ignition (K)	Heat of Reaction[1] ($J\ g^{-1}$)	Density[2] ($g\ cm^{-3}$)	Average Burning Rate[3] ($mm\ s^{-1}$)
SK-338	585	1096.2	1.38	0.46
SK-354	>623	1096.2	1.45	[4]
SK-356	609	1719.6	1.41	0.23
PN758M Blue	467	1355.6	1.51	0.37
PN759 Green	473	1079.5	1.49	0.40
PN757 Red	469	1276.1	1.48	0.37
PN760 Orange	467	1066.9	1.50	0.34
M-18 Green	471	1083.7	1.52	0.26
M-18 Red	449	866.1	1.73	0.29
M-18 Violet	503	991.6	1.70	0.29
M-18 Yellow	445	686.2	1.83	0.34

1 Average of two measurements.
2 Average of two samples.
3 Average for two sets of tests at ambient temperature.
4 Did not burn.

3.2.2 Performance Testing

(a) Burning Rate:

The average burning rate was determined using the procedure outlined in section 3.1.2(a). The results are presented in Table 13.

3.2.3 Safety Testing

(a) Temperature of Ignition:

The temperature of ignition of the smoke compositions was determined using a Wood's - metal bath technique. The sample, mass 0.2 g, was placed in a pyrex test tube and then immersed in a Wood's-metal bath. The bath temperature, initially at 373 K, is increased at the rate of 10 K min^{-1} to a maximum of 633 K or until ignition occurred. The results are given in Table 13.

3.3 Australian M42F1 Percussion Primer

3.3.1 Ignition Effectiveness

The ignition effectiveness of both M42 and M42F1 percussion primers was determined using the go/no-go Bruceton staircase method. The distance corresponding to 50% probability of ignition of a pyrotechnic acceptor was evaluated in an adjustable-length flash tube shown in Figure 10. Forty tests were carried out in this 12.7 mm ID vented tube, made from a steel pipe. Primer caps were placed in the top lid while cambric disks (17 mm OD, 0.2 g), being the acceptor pyrotechnic, were located at the other end of the tube. The cambric sheets were prepared by coating a "J-Cloth" with SR-252 composition [2] mixed in a binder solution of plyobond-acetone-varnish. Disks were punched from dry sheets. The M42 primer caps were from batch WCC-1-950G (Olin Corporation/Winchester Group) and their composition along with that of the M42F1 primer is given in Appendix 4. Table 14 gives the standoff distance for 50% probability of ignition of the SR252 by both the M42 and the M42F1 primers in the configuration considered.

TABLE 14
Standoff Distance

Percussion Primer	Standoff Distance (mm)	Std Dev. σ (mm)	d/σ [1]
M42	97	25	0.5
M42F1	137	53	0.5

[1] d: Step in Bruceton Method.

3.3.2 Pressure Measurement

Comparative tests of the pressure generated by the gas evolved by the M42 and M42F1 (gasless) primer caps were carried out. Both primers were initiated in a closed volume of 1.5 cm^3. A Kistler 701 piezoelectric pressure transducer using a Kistler 5007 charge amplifier was connected to a Nicolet oscilloscope (Model 206) in order to register pressure evolved as a function of time. A total of 4096 measurements were recorded, one every 500 μs over a test duration of about 2 s. Ten tests of each primer were carried out and average results are presented in Table 15. Pressure/time records for both primers are shown in Figure 11.

TABLE 15

Maximum Pressure for M42 and M42F1 Primers

Percussion Primers	Pressure (MPa)			Average[1] Time (ms)
	Min.	Mean[1]	Max.	
M42	5.23	6.03	7.10	0.6
M42F1	0.68	0.81	0.94	12.0

1 Average of 10 readings.

3.4 Phase 2: DSC/DTA on UK Delay SR37

The effect of changes in the sample weight and the heating rate in the DSC or DTA analysis of SR37 was studied in Phase 2. The results are shown in Table 16 and representative traces are presented in Figure 12.

TABLE 16

Effect of Sample Mass on the DTA Characteristics of the Exotherms Associated with the Thermal Decomposition of SR-37

Sample Mass (mg)	Heating Rate (K min^{-1})	Temperature (K)				Residue (mg)
		Exotherm 1		Exotherm 2		
		T_{onset}	T_{max}	T_{onset}	T_{max}	
10.23	10	745	802	824	883	10.30
10.06	10	761	797	829	897	10.09
5.10	10	759	805	824	879	4.36
4.94	10	757	805	827	893	5.16
5.00	5	732	800	822	889	-
2.00	10	777	805	827	885	2.08
2.00	10	774	807	829	888	1.90

4. RESULTS OF UK TESTING

4.1 UK and US Delay Compositions

The mixed compositions were dried at approximately 373 K for 3 hours and then stored over silica gel during testing.

4.1.1 Thermal Characterization

(a) Thermogravimetry:

This technique was used to measure the low temperature mass loss of the compositions when heated to 623 K. Samples of 50 mg were heated at 50 K min^{-1} under flowing argon (25 ml min^{-1}) on a TG 761 thermobalance. The mean percentage mass loss at various temperatures are given in Table 17.

TABLE 17

Percentage Mass Loss of UK and US Delays on Heating

Composition	Mass Loss (%)		
	373 K	573 K	623 K
SR37	0.21 ± 0.03	0.28 ± 0.05	0.31 ± 0.06
SR38	0.14 ± 0.01	0.19 ± 0.02	0.22 ± 0.02
IHM-WD-9-85			0.06 ± 0.01
IHM-WD-3-85			0.09 ± 0.02
IHM-TV-2-85			0.25 ± 0.03
IHM-BD-10-85			0.07 ± 0.01
IHM-BD-11-85			0.11 ± 0.02

(b) Ignition Differential Thermal Analysis (DTA):

The temperature of ignition was determined using a purpose-built DTA apparatus designed to promote ignition in a wide variety of pyrotechnic systems. (The apparatus is described in Appendix 6 and in [3,4]). Samples (50 mg) were compacted into vitreous silica crucibles and heated at 50 K min^{-1} under flowing argon(400 ml min^{-1}) or static air. The results are shown in Table 18.

TABLE 18

Ignition Temperature of UK and US Delay Compositions in Air and Argon

Conditions	Composition	Ignition Temperature (K)	Mean ± sd (K)
Argon	SR37	749, 747, 746	747.3 ± 1.5
	SR38	860, 859, 860	859.7 ± 0.6
Air	SR37	887, N[1], 871, 856, 802	854 ± 37[2]
	SR38	883, 883, 887	884 ± 2
Argon	IHM-WD-3-85	745, 744, 747	745 ± 2
	IHM-WD-9-85	772, 774, 771	772 ± 2
	IHM-TV-2-85	784, 776[3], 784, 781	783 ± 1
	IHM-BD-10-85	1020, 1018, 1018	1019 ± 1
	IHM-BD-11-85	1014, 1012, 1002[2] 1017	1014 ± 3

1 No ignition.
2 Omitting "no ignition" result.
3 Omitted from mean.

(c) Non Ignition Differential Thermal Analysis (DTA)/Non Ignition Differential Scanning Calorimetry (DSC):

This technique consists of investigating the thermal properties of the compositions using a standard DTA or DSC unit under experimental conditions chosen to avoid ignition. DTA on the UK delays was carried out using a Stanton Redcroft 674 DTA Unit. All runs were conducted using quartz crucibles (8 mm x 5 mm) and an atmosphere of flowing argon. Non-ignition conditions were used with a heating rate of 10 K min^{-1} and a sample weight of 25 ± 1 mg. Calcinated alumina was used as a reference standard and certified (ICTA) potassium chromate as a calibrant.

The DTA curves for the UK delays are produced in Figure 13 and the values of peak temperatures are given in Table 19.

All the US delays were run on a Stanton Redcroft 1500 high temperature DSC unit using platinum crucibles, 25 ± 1 mg sample weight, 50 ml min^{-1} flow rate (through DSC head) of argon and a heating rate of 10 K min^{-1}. Before carrying out an experiment on the DSC 1500 all of the samples were run on a DTA 674 to confirm that ignition did not occur. This was to avoid damaging the DSC 1500 which is less robust that the DTA 674 but is preferable when precision work is required. Similar experimental conditions were used on both instruments except a higher flow rate, 100 ml min^{-1}, is required for efficient purging on the DTA 674. The DSC curves for the US delays are shown in Table 20 and Figure 14.

In one case, for sample IHM-BD-11-85, ignition occurred even when the sample size was reduced to 5 mg so this sample was not run on the DSC and the results, determined on the DTA 674 using a 25 mg sample weight, are reported in Table 21. The non ignition DTA curve obtained is shown in Figure 15.

TABLE 19

Peak Temperature for UK Delays from Non-Ignition DTA

Composition	Peak Temperature (K)			
	1	2	3	4
SR37	803	894	921	1023
	806	892	921	1022
	807	894	921	1022
	Mean:	Mean:	Mean:	Mean:
	805 ± 2	893 ± 1	921 ± 0	1022 ± 1
SR38	799	910	956	-
	799	911	960	-
	799	907	954	-
	Mean:	Mean:	Mean:	
	799 ± 0	909 ± 2	957 ± 3	

TABLE 20

Non-ignition DSC Peak Temperature for US Delays

Composition	Peak Temperature (K)		
	1	2	3
IHM-WD-9-85	771 768 772 Mean: 770 ± 2	843 838 846 Mean: 842 ± 4	985 983 982 Mean: 984 ± 2
IHM-WD-3-85	774 773 773 Mean: 773 ± 1		1000 1000 1003 Mean: 1002 ± 2
IHM-TV-2-85	800 796 785 Mean: 793 ± 8		996 995 966 Mean: 986 ± 20
IHM-BD-10-85	1042 1039 1040 Mean: 1036 ± 2		

TABLE 21

Ignition DTA Results for US Delay IHM-BD-11-85

Ignition Temperature (K)			Mean ± s.d.
1	2	3	
1036	1038	1038	1037 ± 1

(d) Combustion Calorimetry (Heat of Reaction):

The exothermicities of the delay compositions were measured by adiabatic combustion calorimetry. The apparatus comprised a Gallenkamp CB110 Autobomb, fitted with a Hewlett-Packard quartz thermometer, with data collection and calculation performed by a CBM

computer. Samples were pressed into pellets using 1 g of composition and a pressing load of 20 MPa. They were tested in triplicate under 1 atmosphere of argon [4]. The results are given in Table 22.

TABLE 22

Exothermicity of UK and US Delay Compositions

Composition	Exothermicity (J g^{-1})			
	1	2	3	Mean
SR37	2350	2385	2335	2360 ± 21
SR38	1858	1891	1820	1858 ± 33
IHM-WD-3-85	866	803	816	828 ± 33
IHM-WD-9-85	1146	1130	1151	1142 ± 13
IHM-TV-2-85	1063	1092	1079	1079 ± 17
IHM-BD-10-85	1439	1423	1427	1443 ± 25
IHM-BD-11-85	2121	2059	2117	2100 ± 33

(e) Evolved Gas Measurements:

The bomb calorimeter was fitted with a pressure measuring system allowing the quantity of gas evolved by the compositions during combustion to be measured. These measurements were made concurrently with the exothermicity determinations (see section d) and are given in Table 23. The figures are adjusted to standard conditions of one atmosphere and 298 K.

TABLE 23

Volume of Gas Evolved by UK and US Delays on Combustion

Composition	Gas evolved (ml g^{-1})			
	1	2	3	Mean
SR37	1.9	2.0	2.2	2.0 ± 0.2
SR38	3.2	3.0	3.2	3.1 ± 0.1
IHM-WD-3-85	2.4	2.8	1.7	2.3 ± 0.6
IHM-WD-9-85	14.1	13.4	13.2	13.6 ± 0.5
IHM-TV-2-85	9.4	7.0	7.0	7.8 ± 1.4
IHM-BD-10-85	3.2	4.1	1.9	3.1 ± 1.1
IHM-BD-11-85	9.5	7.0	9.4	8.6 ± 1.4

4.1.2 Performance Testing

(a) Time to Ignition/Infrared Heating:

In this test, 20 mg samples of each composition were heated by a focussed radiant heat source (tungsten filament) giving exceptionally rapid rates of temperature rise. The samples were pressed into 5 mm diameter discs at 2000 kg load. The time to ignition was measured when the samples, in inconel crucibles, were heated under 75 ml min^{-1} flowing argon and a lamp voltage of 110 V. Results are shown in Table 24. A more detailed description of this test is given in Appendix 7 and references [4,5].

TABLE 24

Ignition Times for UK Delay Compositions under IR Heating

Composition	Time to ignition (s)					
	1	2	3	4	5	Mean
SR37	10.4	10.8	12.3	11.5	11.2	11.2 ± 0.7
SR38	13.7	10.7	13.8	10.4	10.0	11.7 ± 1.9

(b) Time to Ignition/Heated Furnace:

In this test a preheated furnace that is purged with flowing argon is placed over the sample and the time taken for the sample to ignite is measured. The results are presented in Table 25. A detailed description of this test is given in Appendix 8 and references [4,5].

(c) Burning Rate:

To determine the burning rate of the delays, each composition was pressed directly into a steel delay body in four separate increments using several pressing loads to give a total column length of 20 mm. Burning times were measured using a digital timer and a photoelectric cell. A more detailed analysis is given in Appendix 9 and the results are presented in Table 26.

TABLE 25

Ignition Times for UK and US Delay Compositions Using a Heated Furnace

Composition	Furnace Temperature (K)	Time to Ignition (s)			Mean ± s.d.
		1	2	3	
SR37	898	66	65	60	64 ± 2
		64	66	65	
SR38	898	70	73	68	71 ± 2
		69	73	72	
IHM-BD-10-85	1023	116	115	112	116 ± 2
		117	119	118	
IHM-BD-11-85	1023	95	103	105	103 ± 4
		102	105	101	
IHM-HM-3-85	798	108	103	105	106 ± 2
		104	105	105	
IHM-WD-9-85	798	137	135	138	135 ± 2
		136	131	134	
IHM-TV-2-85	798	117	115	120	116 ± 3
		116	111	115	

TABLE 26

Burning Rates of UK and US Delay Compositions

Composition	Burning Rate (mm s^{-1})			
	Pressing Load (MPa)			
	77.2	154.4	231.6	308.8
SR37	15.45	15.08	14.22	13.33
SR38	6.28	5.90	5.68	5.34
IHM-WD-9-85	0.998	0.980	1.007	1.038
IHM-WD-3-85	4.138	4.282	4.394	4.382
IHM-TV-2-85	2.228	2.392	2.336	2.390
IHM-BD-10-85	7.75	7.91	7.00	6.33
IHM-BD-11-85	37.37	36.17	32.86	31.01

(b) *Gap Test/Standoff Test:*

This test measures the ignitability of pyrotechnic compositions to different type of thermal input by measuring the distance between an igniter and an acceptor such that 50% probability of ignition of the acceptor is achieved. Two sets of experiments were carried out. The first set used the RARDE Gap Ignition Test apparatus (see Appendix 10) whilst for the second set, an 'Anglicised' version of the Australian Standoff Tube was manufactured, in house, at RARDE. Details of the Standard Australian Standoff Test are given in Appendix 3.

With the RARDE Gap Ignition test, SR37 and SR38 pressed at two different loads were used as acceptor pellets and SR44 [2] was used as the donor. With the 'Anglicised' Standoff test, the gasless M42F1 Percussion primers, (Appendix 4) supplied by Australia, were used as the donors and SR37, SR38 and SR44 as acceptors. Table 27 presents the results of the Bruceton analysis used to determine the gap for 50% ignition of the UK delays as acceptors.

4.1.3 Safety Testing

The UK mandatory explosive powder safety tests were carried out by RARDE Waltham Abbey on the delay compositions SR37 and SR38. The results are given in Table 28. Detailed descriptions of the tests are given in Appendix 5.

TABLE 27

Gap Test Results for UK Delays at Various Pressing Loads

Acceptor Composition	Pressing Load	Gap for 50% Ignition (cm)	
		RARDE Gap Ignition (SR44 donor)	'Anglicised' Standoff Tube (M42F1 donor)
SR37	77.2 MPa	5.90	-
	231.6 MPa	5.49	-
	105.8 MPa	-	24.7
SR38	77.2 MPa	4.93	-
	231.6 MPa	5.55	-
	105.8 MPa	-	3.92
SR44	77.2 MPa	8.9-4.3	-
	105.8 MPa	-	22.86

TABLE 28

Safety Test Results for UK Delay Compositions

Test	Composition	
	SR37	SR38
Figure of Insensitiveness	110	200
Mallet Friction Wood on Wood (%) Steel on Steel (%)	 0 100	 0 50
Rotary Friction	3.3	3.8
Temperature of Ignition	Nothing under 673 K (sample decomposing)	Nothing under 673 K (sample decomposing)
Bickford Fuze Test	Ignites and burns vigorously	Ignites and burns quietly (2nd attempt)
Train Test	Ignites and supports train vigorously throughout	Ignites and supports train steadily throughout
Electrostatic Test	20,000 J at 1000 μ F	Fired at 4.5 joules (1st) Fired at 0.45 joules (1st) No ignitions at 0.045 joules

4.2 Canadian, UK and US Smoke Compositions

Samples of the 4 UK smoke compositions were received from Royal Ordnance Factory, Glascoed. The 3 Canadian smoke compositions were received as machined blocks of castable smoke and samples were simply cut out from the blocks as required. No samples of the US compositions were received. Sample preparation was as detailed in Appendix 1 and all samples were stored over silica gel during testing.

4.2.1 Thermal Characterisation

(a) Ignition Differential Thermal Analysis (DTA):

Samples were compacted into silica crucibles and heated, at 50 K min^{-1}, in static air. The procedure is detailed in Appendix 6 and references [3,4]. The results are presented in Table 29.

TABLE 29

Temperature of Ignition of Canadian and UK Smoke Compositions

	Canadian Compositions			UK Compositions			
	SK338	SK354	SK356	PN757	PN758M	PN759	PN760
Temperature of Ignition (K)	499.2	615.2	502.3	503.3	483.1	491.1	481.3

(b) Non Ignition Differential Thermal Analysis (DTA):

Approximately 20 mg samples of each of the seven smoke compositions were analysed using a Stanton Redcroft DTA 674. The samples were placed in open aluminium pans using an atmosphere of flowing nitrogen (200 ml min^{-1}) and a heating rate of 10 K min^{-1}. Silicon was used as a reference standard. Typical DTA curves are reproduced in Figures 16 and 17 and the values of the peak endotherm temperatures are given in Table 30.

TABLE 30

Non-Ignition DTA Endotherm Temperatures for Canadian and UK Smoke Compositions

Composition	Peak Temperature (K)		
	1	2	3
SK338		419.1	
SK354		421.8	
SK356		420.6	
PN757	390 (s)	417.1	441
PN758M	391 (s)	420.3	-
PN759	391 (s)	421.1	459.6
PN760	388 (vs)	422.0	438 (s)

(s) small
(vs) very small

4.2.2 Performance Testing

No tests were conducted in this area.

4.2.3 Safety Testing

The temperature of ignition of the UK smoke compositions determined using the standard safety test (see Appendix 5) are shown in Table 31.

TABLE 31

Temperature of Ignition of UK Smoke Compositions

	Composition			
	PN757	PN758M	PN759	PN760
Temperature of Ignition (K)	464	463	468	463

4.3 *Phase 2: DSC/DTA on UK Delay SR37*

The samples were investigated using a Stanton Redcroft DSC 1500 high temperature DSC apparatus. (Appendix 11). The samples were run in platinum crucibles 4 mm in height, 6mm diameter, in an atmosphere of argon flowing directly through the head at 50 ml min^{-1}. Argon was also passed through the furnace chamber itself as a blanket gas.

Sample weights of 5, 10, 15 and 25 mg were chosen and run at heating rates of both 5 K min^{-1} and 10 K min^{-1}. A minimum of duplicate runs was carried out in all cases and calibration runs were carried out at regular intervals using the phase change of potassium chromate in the region of 943 K.

The results at 5 K min^{-1} and 10 K min^{-1} are summarised in Tables 32 and 33 respectively. The peak numbers used in this tables refer to the notation used by Charsley and Ottaway [6].

TABLE 32

DSC Peak Temperatures for SR 37 Heated at 5 K min^{-1}

Sample Mass (mg)	Peak Temperatures (K)			
	1	2	3	4
5 ± 1	780	883	905	1033
	783	890	908	1033
Mean	781 ± 2	887 ± 4	907 ± 2	1033 ± 0
10 ± 1	784	883	909	1009
	784	878	907	1004
	784	879	907	1009
Mean	784 ± 0	880 ± 2	908 ± 1	1007 ± 2
15 ± 1	786	878	909	Peak not resolved
	793	884	914	(1005)[1] 1011
Mean	790 ± 4	881 ± 3	911 ± 3	
25 ± 1	786	880	908	(994)[1] 1003
	788	881	910	(994)[1] 1003
Mean	787 ± 1	881 ± 1	909 ± 1	994 ± 0 1033 ± 0

1 Minor features given in brackets.

TABLE 33

DSC Peak Temperatures for SR 37 Heated at 10 K min^{-1}

Sample Mass (mg)	Peak Temperatures (K)			
	1	2	3	4
5 ± 1	802	890	923	1038
	803	888	919	1034
Mean	803 ± 1	889 ± 1	921 ± 2	1036 ± 2
10 ± 1	805	884	917	920 (930)[1]
	805	888	921	929 (935)[1]
Mean	805 ± 0	886 ± 2	919 ± 2	924 ± 4 (933 ± 3)
15 ± 1	803	889	923	(925)[1] 933
	803	886	918	(919)[1] 930
Mean	803 ± 0	888 ± 2	921 ± 3	(922 ± 3) 932 ± 2
25 ± 1	797	879	914	(1006)[1] 1017
	796	881	914	(1007)[1] 1019
Mean	797 ± 1	880 ± 1	914 ± 0	(1007 ± 1) 1018 ± 1

1 Minor features given in brackets.

5. RESULTS OF US TESTING

5.1 UK and US Delay Compositions

5.1.1 Thermal Characterization

(a) Differential Scanning Calorimetry (DSC):

DSC analysis was conducted using a Du Pont 1090-B DSC operating in the non-isothermal mode with a high pressure DSC cell. The operating conditions used for the experiments are as follows:

UK Compositions: 2.5 mg sample, heating rate 20 K min^{-1} under flowing nitrogen at 3.45 MPa.

US Compositions: 1-2 mg sample, heating rate 10 K min^{-1} under flowing nitrogen or air

Potassium nitrate was used as the temperature calibration material.

Typical DSC traces for all the pyrotechnic delay compositions are shown in Figures 18 and 19. Analysis in terms of the positions of the important exotherms and endotherms are given in Table 34. These results were for a single analysis and the temperature range covered was 300 K to 820 K.

TABLE 34

DSC Results for UK and US Delays

			Peak 1			Peak 2		
Composition	Gas	Sample Mass (mg)	T_{onset} (K)	T_{max} (K)	ΔH ($J\ g^{-1}$)	T_{onset} (K)	T_{max} (K)	ΔH ($J\ g^{-1}$)
SR37	N_2	2.5	524.9	526.6	-1.8			
SR38	N_2	2.6	523.5	526.0	-1.0			
IHM-BD-10-85	Air	2.08	-	-	-			
IHM-BD-10-85	N_2	1.16	-	-	-			
IHM-BD-11-85	Air	1.69	-	-	-			
IHM-BD-11-85	N_2	2.05	-	-	-			
IHM-TV-2-85	Air	1.35	-	574.0	-	-	775.0	-
IHM-TV-2-85	N_2	1.47	-	573.2	-	-	780.7	-
IHM-WD-3-85	Air	1.35	-	573.0	-	746.1	767.2	-239.7
IHM-WD-3-85	N_2	1.21	-	573.0	-	750.7	764.9	-160.7
IHM-WD-9-85	Air	1.13	-	574.0	-	-	772.0	-
IHM-WD-9-85	N_2	1.31	-	573.2	-	-	775.7	-

(b) Heat of Explosion:

The heat of explosion of the delays was determined using a Parr Bomb Calorimeter pressurized with argon to 25 atm (Ref [7]). A B/KNO_3 igniter (0.5 g) was used to initiate the compositions as the standard hot wire igniter was insufficient to achieve ignition. The heat output of the B/KNO_3 igniter was subtracted from the total heat output to give the true heat of explosion (Table 35). The results are the average for duplicate analyses.

TABLE 35

Heat of Explosion for UK and US Delay Compositions

Composition	Heat of Explosion ($J\ g^{-1}$)
SR37	-
SR38	-
IHM-BD-10-85	Sample melted, did not burn.
IHM-BD-11-85	Sample melted, did not burn.
IHM-TV-2-85	1140.2
IHM-WD-3-85	822.2
IHM-WD-9-85	1376.5

5.1.2 *Performance Tests*

(a) Burning Rate:

The burning rate of the delay compositions was determined by pressing several increments of the composition into a 22.5 mm aluminium column at 207 MPa. The column diameter was 5.1 mm for both IHM-BD-10-85 and IHM-BD-11-85, whilst a 6.6 mm diameter column was used for the rest. An ignition increment was pressed onto the top of the delay and the system ignited with an M-100 matchead igniter. The tests were conducted at 294 K and the burning times were measured electronically. Table 36 presents the average of 14-20 separate tests. More detailed test information is given in Appendix 12.

TABLE 36

Burning Rates of UK and US Delay Compositions

Composition	Burning Rate ($mm\ s^{-1}$)
SR37	15.94
SR38	-
IHM-BD-10-85	7.33
IHM-BD-11-85	34.65
IHM-TV-2-85	-
IHM-WD-3-85	5.98
IHM-WD-9-85	1.26

5.1.3 Safety Testing

The sensitivity of each composition to friction, impact and electrostatic discharge was determined using the U.S. friction test, drop test and electrostatic discharge test (ESD) respectively. The impact test uses a 5 kg mass being dropped vertically, from various heights, onto the sample under test. The friction test uses a weighted pendulum, which impacts a sliding block. The sample is placed between the sliding block and a stationary wheel and decomposes due to the movement of the sliding block. The pendulum speed was 24.7 ms^{-1}. The ESD test measures the minimum energy required to initiate a sample when placed between a steel needle and a metal test plate. The needle is connected to a capacitor bank and the power supply is a 500 V DC source. Detailed descriptions of these tests are given in Appendix 13. Table 37 shows the safety data for the delay compositions.

TABLE 37

Sensitivity/Safety Data for UK and US Delay Compositions

Composition	Impact (mm)	Friction (kg)	ESD (J)
SR37	600	18.1	0.0200
SR38	-	-	-
IHM-BD-10-85	600	444	0.125
IHM-BD-11-85	600	2.3	0.00875
IHM-TV-2-85	600	444	0.500
IHM-WD-3-85	600	444	0.125
IHM-WD-9-85	600	444	0.100

5.2 Canadian, UK and US Smoke Compositions

5.2.1 Thermal Characterization

(a) Differential Scanning Calorimetry (DSC):

DSC analysis was conducted on all the smoke compositions using the procedure as outlined in Section 5.1.1(a). The gas used was nitrogen and duplicate samples were analysed. Separate results for each of the duplicate analyses are presented in Table 38. Representative DSC traces are shown in Figures 20, 21 and 22.

TABLE 38

DSC Results for Canadian, UK and US Smoke Compositions

Composition	Mass (mg)	Exotherm 1			Exotherm 2		
		T_{onset} (K)	T_{max} (K)	ΔH (J g^{-1})	T_{onset} (K)	T_{max} (K)	ΔH (J g^{-1})
US Coloured Smokes							
M-18 Yellow	4.25/4.64	481.1/465.2	487.3/484.4	837.6/1059.4			
M-18 Red	3.61/3.49	481.1/482.8	506.8/514.4	968.2/1192.0	587.8/574.5	617.2/606.7	293.7/472.4
M-18 Green	5.30/3.33	473/471.2	487.7/484.2	599.1/ 951.4			
M-18 Violet	4.44/4.42	488.5/505.6	499.5/516.6	1047.3/ 546.0			
UK Coloured Smokes							
PN758M	4.53/4.63	469.2/469.9	481.7/482.7	1345.6/1412,5	591.1,588.1	657.7,639.4	292.5/335.6
PN759	4.41/4.40	457.9/457.4	457.2/456.8	1230.9/1275.7	594.0,600.5	649.3,651.0	604.2/528.0
PN757	4.61/4.71	473.2/470.6	496.7/489.4	1668.9/ 828.4	593.4,558.9	628.4,675.5	311.3/470.3
PN760	4.83/4.70	454.2/456.3	472.3/472.2	1551.4/1631.0	590.1,621.3	643.9,668.9	324.3/703.7
Canadian Coloured Smokes							
SK354	4.69	474.7	489.3	711.7	605.8	621.1	110.9
SK356	4.77/4.65	473.4/473.7	494.3/497.7	835.9/ 306.7			
SK338	4.66/4.85	469.1/471.2	490.6/491.5	1188.7/1109.2			

5.3 Australian M42F1 Percussion Primer

5.3.1 Sensitivity

In evaluating the M42F1 percussion primer, its performance was compared to that of the PVU-1/A primer, a U.S. Navy version of the M42-5086. Bruceton and rundown tests were performed on the M42F1 primers to determine sensitivity. They were found to be comparable to the PVU-1/A, with a Bruceton all-fire (M+5S) of 12.03 m-g and a rundown all-fire (M+5S) of 15.35 m-g Table 39 gives details of these results.

TABLE 39

Bruceton and Run-Down Sensitivity Analysis of M42F1 Percussion Primer

Bruceton Analysis

Test Date: 9-8-86	Ball WT: 55.0 g
Primer: M42F1	Mean HT: 163.1 mm
Lot: N/A	Std Dev: 11.2 mm
Test: Bruceton	

All-fire (M+5S): 218.9 mm ; 12.03 m-g
All-fire (M+3S): 196.6 mm ; 10.81 m-g
No-fire (M-2S): 140.7 mm ; 7,73 m-g

Run-down Analysis

Test Date: 9-8-86	Ball Weight: 55.0 g
Primer: M42F1	HT @ 50% Funct: 144.8 mm
Lot: N/A	Std Deviation: 26.9 mm

All-fire (M+5S): 279.1 mm ; 15.35 m-g
All-fire (M+3S): 225.3 mm ; 12.40 m-g
No-fire (M-2S): 90.9 mm ; 5.00 m-g

No. Tested @ ea. HT: 25

Height	Misfires
3	25
4	23
5	22
6	7
7	1
8	2
9	0

5.3.2 Differential Scanning Calorimetry (DSC)

The primer composition was extracted and a DSC analysis was conducted. The DSC was a Du Pont 9900 and the analysis was carried out in static air at a heating rate of 10 K min^{-1}. Figure 23 presents a typical DSC trace.

5.3.3 Energy Output

Energy output analysis was performed using a McDonnell Aircraft Co. honeycomb cell output test fixture [8]. The fixture measures energy output by mechanical work performed by pressure so it was expected that the M42F1 primers would have a low output being essentially gasless. As anticipated, the mean energy output was 11.52 mm-kgf. The mean energy output for a PVU-1/A primer is 633.7 mm-kgf.

5.3.4 Pyrotechnic Delay Time

The M42F1 primers were loaded into delay cartridges to determine how the low pressure output would affect pyrotechnic delay burn time in an obturated cartridge at 294 K. The primers were loaded into M93 delay cartridges (Figure 24) using a T-10 delay composition with 4.5% boron and 95.5% barium chromate. The same length of delay was used for both primer types (i.e., column length was optimized to give the proper delay time for the PVU-1/A primer only). The specified delay time for these cartridges must be a minimum of 800 ms and a maximum of 1200 ms. The delay time for the cartridges loaded with M42F1 primers ranged from 992 ms to 1248 ms with a mean of 1187.8 ms and standard deviation of 71.0 ms. Delay time for control cartridges loaded with PVU-1/A primers ranged from 1077 ms to 1213 ms with a mean of 1107.9 ms and a standard deviation of 13.8 ms.

6. ACKNOWLEDGEMENTS

Clearly a project of this size and scope involved many more people than the five who appear as authors. The authors would therefore like to thank those, in all the laboratories, who provided the technical assistance in carrying out the many tests and also those who provided support and information for this project.

7. REFERENCES

1. de Yong, L.V. and Wanat, E., (1986). A Standoff Test to Determine the Ignitability of Pyrotechnics and the Ignition Effectiveness of Pyrotechnic Igniters. *Report MRL-R-998,* Materials Research Laboratories, Melbourne, Australia.

2. Cackett, J.C. (1981). The Properties of SR Pyrotechnic Compositions, *Report 1/71.* Royal Armament Research and Development Establishment, Fort Halstead, Kent, U.K.

3. Charsley, E.L., Ottaway, M.R., Cox C.T., Barton, T.J., and Jenkins, J.M., (1982). Investigation of the Ignition Temperature of Pyrotechnic Systems Using a Purpose Designed Differential Thermal Analysis Apparatus, *Thermochimica Acta,* 52, 321-332.

4. Lindsley, G.I., Robinson, E.A., Charsley, E.L. and Warrington, S.B. (1986). A Comparison of the Ignition Characteristics of Selected Metal-Oxidant Systems, *Proceedings of the 11th International Pyrotechnics Seminar, Vail, Colorado,* pp 425.

5. Robinson, E.A., Lindsley, G.I., Charsley, E.L. and Warrington, S.B., (1985). Assessing the Ignition Characteristics of Pyrotechnics, *Proceedings of the 16th ICT Combined with 10th International Pyrotechnics Seminar, Karlsruhe, Germany,* July 2-5, 1985, p 25-1 (Fraurhofer - Institut fur Treib-und Explosivstoffe, Karlsruhe)

6. Charsley, E.L. and Ottaway, M.R. (1977). Thermal Studies on the Boron-Molybdenum Trioxide Pyrotechnic Delay System. *In, Reactivity of Solids,* pp 737-741 (Plenum, New York). J. Wood (ed)

7. US Military Specification MIL-STD-286.

8. Schimmel, M.L. and Drexelius, V.M. (1967). Measurement of Explosive Output. *The Proceedings of the 5th Symposium on Electroexplosive Devices, Philadelphia, USA, June; 1967,* pp 5.1-5.20 (The Franklin Institute, Philadelphia, Pa).

APPENDIX 1

DETAILS OF EXCHANGE MATERIALS

1. UK Delays: These materials were supplied as separate ingredients which were either sieved mixed (3 x BSS240 sieve) or tubular mixed together to make the following compositions:

Composition	Ingredients	Proportion by Weight (%)
1. SR 37	Boron (amorphous)	11 ± 0.3
	Molybdenum Trioxide	89 ± 0.3
2. SR 38	Boron (amorphous)	7 ± 0.3
	Molybdenum Trioxide	93 ± 0.3

2. UK Smokes: All laboratories were supplied with 4 off each L46A1, L47A1, L48A1 and L49A1 coloured smoke grenades. These were broken down, the smoke composition extracted, crushed lightly, then sized through a BSS 18 sieve.

Composition	Ingredients	Proportion by Weight (%)
1. PN757 (L48A1-Red)	Lactose (monohydrate)	29
	Potassium Chlorate	30
	Dye, CI disperse red 9	39
	Kaolin	2
2. PN758M (L46A1-Blue)	Lactose (monohydrate)	23
	Potassium Chlorate	26
	Dye, CI disperse blue 150	48
	Zinc Oxide	3
3. PN759 (L47A1-Green)	Lactose (monohydrate)	25
	Potassium Chlorate	25
	Dye, CI solvent green 3A	29
	Dye, CI solvent yellow 33	18
	Zinc Oxide	3
4. PN760 (L49A1-Orange)	Lactose (monohydrate)	25
	Potassium Chlorate	26
	Dye, CI solvent yellow 33	28
	Dye, CI disperse orange 11	18
	Zinc Oxide	3

3. U.S. Delays: These materials were supplied pre-mixed. Each composition was passed through a BSS 18 sieve to remove any agglomerates that may have formed during shipment.

Composition	Ingredients	Proportion by Weight (%)
1. IHM-WD-9-85	Tungsten	31
	Barium Chromate	54
	Potassium Perchlorate	10
	Diatomaceous Earth	5
2. IHM-WD-3-85	Tungsten	55.5
	Barium Chromate	34.5
	Potassium Perchlorate	5
	Silicon Dioxide	5
3. IHM-TV-2-85	Tungsten	44
	Barium Chromate	43
	Potassium Perchlorate	7
	Silicon Dioxide	5
	Viton A	1
4. IHM-BD-10-85	Boron	4
	Barium Chromate	96
5. IHM-BD-11-85	Boron	10.3
	Barium Chromate	89.7

4. U.S. Smokes: All laboratories were supplied with 4 of each M-18 Green, Red, Violet and Yellow smoke grenades. These were broken down, the smoke composition extracted, crushed lightly then sized by passing through a BSS18 sieve.

5. Canadian Smokes: These compositions were supplied as machined blocks of castable smoke composition.

Composition	Ingredients	Proportion by Weight (%)
1. SK-338 (Red)	Dye, 1-MAAQ	41
	Potassium Chlorate	25
	Sodium Bicarbonate	6
	Sulphur	8
	Binder	20
2. SK-354 (Orange)	Dye,1-AAQ	46.00
	Potassium Chlorate	25.30
	Lactose	8.45
	Sodium Bicarbonate	2.25
	Binder	18.00
3. SK-356 (Orange)	Dye, 1-AAQ	30
	Potassium Chlorate	38
	Lactose	12
	Sodium Bicarbonate	2
	Binder	18

6. Australian Percussion Primers: All laboratories were supplied with 500 M42F1 gasless percussion primers. The composition of these was:

Ingredients	Proportion by Weight (%)
Boron	9.5
Red Lead Oxide	85.5
Tetracene	5.0

APPENDIX 2

SUMMARY OF TEST PROGRAMME FOR ALL EXCHANGED MATERIALS

Compositions	Thermal Characterization	Performance Tests	Safety Tests
UK Delays			
SR37	AUS: DSC	AUS: Standoff Test	AUS: F of I, T of I, Electrostatic
SR38	CANADA: DSC (SR37 only) Combustion Calorimetry	CANADA: Burning Rate	CANADA: T of I
	UK: Thermogravimetry Ignition DTA Non Ignition DTA/DSC Combustion Calorimetry Evolved Gas	UK: Burning Rate Gap/Standoff Test Time to Ignition/Heated Furnace	UK: F of I, T of I, Electrostatic Mallet and Rotary Friction Train Test Bickford Fuze
	US: DSC	US: Burning Rate (SR37 only)	US: Electrostatic, Sliding Friction Impact (Drop Test)

Compositions	Thermal Characterization	Performance Tests	Safety Tests
US Delays			
IBM-WD-3-85	AUS: DSC	AUS: Standoff test	AUS: F of I, T of I, Electrostatic
IBM-WD-9-85	CANADA: DTA (TV-2-85, WD-3-85, BD-11-85 only)	CANADA: Burning Rate	CANADA: T of I
IBM-TV-2-85	Combustion Calorimetry	UK: Burning Rate	UK: ---
IBM-BD-10-85	UK: Thermogravimetry	Time to Ignition/Heated Furnace	US: Electrostatic
IBM-BD-11-85	Igaition DTA	US: Burning Rate	Sliding Friction
	Non Ignition DSC		Impact (Drop Test)
	Combustion Calorimetry		
	Evolved Gas		
	US: DSC		
	Combustion Calorimetry		

Compositions	Thermal Characterization	Performance Tests	Safety Tests
Canadian Smokes			
SK-338	AUS: ---	AUS: ---	AUS: ---
SK-354	CANADA: DTA (SK338 only) Combustion Calorimetry	CANADA: Burning Rate	CANADA: T of I
SK-356	UK: Ignition DTA Non Ignition DTA	UK: ---	UK: ---
	US: DSC	US: ---	US: ---
US Smokes			
M-18 Green	AUS: DSC	AUS: Standoff Test	AUSD: T of I, F of I
M-18 Red	CANADA: DTA/TGA (M-18 Red only) Combustion Calorimetry	CANADA: Burning Rate	CANADA: T of I
M-18 Violet	UK: ---	UK: ---	UK: ---
M-18 Yellow	US: DSC	US: ---	US: ---

Compositions	Thermal Characterization	Performance Tests	Safety Tests/
UK Smokes			
PN757 (L48A1)	AUS: DSC	AUS: Standoff Test	AUS: T of I, F of I
PN758M (L46A1)	CANADA: DTA/TGA (PN757 only) Combustion Calorimetry	CANADA: Burning Rate	CANADA: T of I
PN759 (L47A1)		UK: ---	UK: T of I
PN760 (L49A1)	UK: Ignition DTA	US: ---	US: ---
	US: DSC		
Australian M42F1 Percussion Primers	AUS: ---	AUS: Pressure/time CANADA: Pressure/time Standoff Test	AUS: F of I, T of I, Electrostatic
	CANADA: ---		CANADA: ---
	UK: ---	UK: Gap Test	UK: ---
	US: DSC	US: Energy Output Delay Burning Time	US: Drop Sensitivity

APPENDIX 3

MRL STANDOFF TEST [1]

The standoff test closely models the gap test that is used for explosive testing. The test involves separating a donor igniter from an acceptor pyrotechnic composition in a fixed diameter metal cylinder. The donor is fired at the acceptor with the assumption that the donor stimulus will vary inversely with the air gap between the donor and the acceptor (the standoff distance). By applying conventional testing techniques and statistics, the probability of initiation of the acceptor is judged by the standoff distance over which the energy required for ignition can be transferred. Altering the donor with a standard acceptor allows variations in the donor's performance to be determined; by altering the acceptor with a constant donor, the ignitability or the ease of ignition of the acceptor can be determined.

Ideally, to determine the ignitability of a pyrotechnic compositions or the ignition effectiveness of an igniter, the distance between the igniter and the pyrotechnic composition over which ignition would be transferred with 100% probability of reliability would be determined. This style of approach is, however, impractical, costly and time consuming due to the large number of tests involved. By using GO/NO GO statistical techniques, as in the Bruceton Method, the distance between the donor and acceptor for 50% probability of ignition of the acceptor can be easily determined using relatively few tests. The distance corresponding to 50% probability of ignition, or the mean standoff distance, determined from 20-30 seperate tests, can then be used to rank the ignition effectiveness of the donor or the ignitability of the acceptor.

The standoff tube used for these experiments is shown in Figure 25. It is a 13 mm ID vented brass tube with the donor being affixed at the top and a pellet of pyrotechnic composition (acceptor) placed inside. The value of standoff distance between donor and acceptor is altered using variable length inserts below the acceptor. The acceptor mass was set at 1.0 g and was pressed into a 13 mm diameter pellet at a pressing load of 8.25 kN (62.1 MPa).

APPENDIX 4

CHEMICAL COMPOSITION OF M42 AND M42F1 PERCUSSION PRIMERS

M42 primer (PA101)	**Weight %**
Basic Lead Styphnate	53.0
Barium Nitrate	22.0
Antimony Sulphide	10.0
Aluminium	10.0
Tetracene	5.0

M42F1 Primer (MRL(X)408)	
Boron (Amorphous)	9.5
Red Lead Oxide	85.5
Tetracene	5.0

APPENDIX 5

UK SENSITIVITY TESTS

Impact (F of I)

The figure of insensitiveness (F of I) is derived from tests in the Rotter Impact Machine in which the pyrotechnic composition and a standard reference explosive (RDX) are alternately subjected to a weight falling from various heights. An ignition is considered to have occurred if over 1ml of gas is generated by the impact of the weight on the explosive or pyrotechnic sample.

Mallet Friction

The sensitiveness to friction of pyrotechnic compositions is tested by striking thin layers, spread on a flat test plate, with swinging, glancing blows from a mallet. Various materials are used for the test plates (Anvil) and Mallet heads. An ignition is considered to have occurred if a crack, spark or flash is produced by the blow. Because of the variable results obtained from the test only 3 levels of response are reported: 0% ignitions, 50% ignitions and 100% ignitions.

Rotary Friction

A machine has been developed to evaluate the sensitiveness of explosives to friction between two surfaces. An explosive sample is either cut as a sliver or spread by means of a spatula so that its thickness is no more than about 0.1 mm on a flat strip of steel. The explosive sample is held under a predetermined load on the strip of steel in stationary contact with the periphery of a wheel, the surfaces having been previously prepared to a specific finish. The wheel is then driven by means of a heavy flywheel and motor at a given velocity through a constant strike length, after which the surfaces are separated. An ignition is usually identified by a flash or audible report, but even a little smoke or blackening of the sample is considered as an ignition for the purposes of the test. The standard procedure at present is a 50 shot up and down run, varying the velocity for each shot.

Results are expressed relative to those for standard RDX by assigning a value of 3.0 to the Figure of Friction for the standard RDX.

Broad sensitiveness categories can be assigned:

Very sensitive	<3
Sensitive	>3 but <6
Comparatively insensitive	>6

Temperature of Ignition (T of I)

The temperature of ignition is determined by raising the temperature of a 0.20 gram sample of the composition at 5 K min^{-1} in a standard apparatus until the temperature reaches 673 K. Any event is noted.

Ignition by Flash (Bickford Fuze Test)

A 3 gram sample of pyrotechnic composition in a test tube is subjected to the short burst of flame emitted from the end of a length of Bickford fuze. Observations are made of the ease and type of ignition.

Train Test

A sample of pyrotechnic composition in a 300 mm long, 12.7 mm wide steel trough is ignited by a luminous gas flame at one end. Observations of the ignition and ability to support the train are made.

Electrostatic Spark Sensitiveness

The sensitiveness to electrical spark is measured by discharging a condenser through a layer of explosive between 2 metal electrodes. The energy in the spark is altered by using condensers of differing capacities. The results are reported as one of the following 4 categories:

a. No ignition at 4.5 joules

b. Ignitions at 4.5 joules but not at 0.45 joules

c. Ignitions at 0.45 joules but not at 0.045 joules

d. Ignition at 0.045 joules - this result will lead to further detailed investigation.

APPENDIX 6

IGNITION DIFFERENTIAL THERMAL ANALYSIS (DTA) APPARATUS [3]

The ignition DTA head, designed to replace the standard Stanton Redcroft DTA-673 head, is shown in Figure 26. The sample and reference materials are contained in flat-bottomed quartz crucibles, C, 6 mm in diameter and 20 mm in length. The crucibles are supported by plate-type chromel-alumel thermocouples, E, fitted with locating ears and having 0.5 mm diameter wire leads. The latter replaced the 0.25 mm diameter wires originally used, which were found to be not sufficiently robust. The thermocouples are supported in short lengths of twin bore alumina rods, A, and passed into a four-bore alumina rise rod, F, on which the DTA block assembly is supported. The DTA block, B, is made from stainless steel and has wells 8 mm in diameter. The removable lid, D, made from the same material, serves to locate accurately the crucibles and to protect the thermocouples from attack by reaction products. It also ensures that the crucibles are effectively isolated from the block, thereby minimising heat losses.

The signal from the sample thermocouple is fed to a temperature indicator (Stanton Redcroft Model 9812) which provides a digital display of the temperature accurate to one degree centrigrade and a linearised temperature output which is recorded on one channel of a two pen strip-chart recorder, the other channel recording the DTA signal. The standard DTA 673 programmer was replaced by a Model 683 unit and transformer, giving linear rates of up to 60 K min^{-1} in normal use, compared with the maximum standard rate of 20 K min^{-1}. Experiments are normally carried out in static air covering the range from ambient to 673 K. For research purposes, when experiments in argon may be required, it is necessary to ensure that the air trapped by the lid does not cause oxidation of the fuel. Although this was initially achieved by evacuation of the unit, followed by flushing with argon, it was later found to be equally effective to place a shallow crucible containing titanium powder on top of the lid and pass argon through the apparatus, at 400 ml min^{-1}, in the normal way.

APPENDIX 7

INFRARED HEATING APPARATUS [4,5]

This unit is based on a focussed radiant source (Research Incorporated model 4141) and is shown in Figure 27. A tungsten filament, F, is focussed by an ellipsoidal mirror, M, to provide an elliptical spot some 8 x 5 mm at a focal length of 38 mm. The maximum incident heat flux is approximately 140 W cm^{-2}. Exceptionally rapid rates of temperature rise are possible, to equilibrium temperatures in excess of 1300 K. The lamp, which is air-cooled, is powered via a mains voltage stabiliser, a variac, and a switched transformer, allowing settings from 20-250 V to be used in steps of a nominal 10 V. The lamp is mounted in a modified microscope stand, enabling accurate height adjustment, and giving good stability of the lamp with respect to the hot-stage below.

The hot-stage microscope unit can be used in its standard form, but for simplicity the present unit contains no furnace. The sample, S, rests in an inconel pan on a plate-type chromel/alumel thermocouple, T, and is heated directly through the quartz window, Q. The hot-stage may be centred accurately be means of the screws, B. Thereafter, by slackening screw, N, it may be removed for cleaning and replaced without need for realignment. Centering is achieved by placing a blackened, oxidised crucible upon the thermocouple, and moving the stage until the maximum equilibrium temperature is reached with the lamp operating at a fixed voltage. Calibration of the unit is performed by selecting power settings of 100 or 200 V, and recording the temperature reached by the oxidised crucible. Adjustments to the variac in the power train are made to bring these recorded temperatures within specification; such fine adjustment is necessary to allow for the ageing of the lamp filament. As with the time to ignition unit (Appendix 8), a standard run is recorded using 50:50 tungsten-potassium dichromate, in the form of 20 mg pellets. An argon flow of 75 ml min^{-1} is maintained through the unit during these runs, and during most other work. Calibration data recorded over a period of 6 months give a time to ignition for the tungsten-potassium dichromate pellets of 9.7 ± 1.1 seconds at a power setting of 80 V.

APPENDIX 8

TIME TO IGNITION APPARATUS [4,5]

An overall view of the unit is shown in Figure 28 together with a larger scale representation of the head. The sample S, contained in a 20 mm long, 6 mm OD quartz crucible rests on a robust chromel/alumel plate thermocouple, T. The discs D and E, and the spacers, I, are constructed from inconel. As well as supporting the crucible, these discs, being a close fit in the furnace tube, provide some protection for the thermocouple from ejected reaction products.

The sample is heated by the water-cooled furnace, F, which may be raised and lowered manually while being restrained by guide wheels. The furnace may thus be quickly and reproducibly lowered to seat upon an 'O' ring, against which it forms a gas-tight seal by virtue of its weight. Gas can be passed into the top of the furnace via the porous ceramic wool plug, P. A fan, not shown in the diagram, quickly cools the head when the furnace is raised at the end of a run. Ignition is recorded as a sharp deviation on the otherwise smoothly rising temperature trace, displayed on a chart recorder. The time to ignition is measured from the recorder trace.

The furnace is controlled by a variable rate programmer, which allows linear rates at up to 20 K min^{-1}, or maintenance of isothermal temperatures to within 0.5 K. The ability to heat the sample linearly is used in checking the thermocouple calibration. Samples of ultra-pure metals are heated at 3 K min^{-1}, and melting detected by an arrest on the recorder temperature trace. The indicated melting points are typically within 0.5 K of the true value. The reproducibility of heating is checked as follows: After temperature calibration, the furnace is set to an isothermal temperature of 773 K with a crucible containing 50 mg of alumina in place. The furnace is then raised and the sample cooled. On lowering the furnace again the heating curve is recorded. The temperature reached by the sample after 30,60,90,120 and 180 seconds is noted and compared with earlier records.

APPENDIX 9

MEASUREMENT OF DELAY BURNING RATE

The sample of delay composition is consolidated directly into steel delay bodies at various pressing loads. Four increments of composition are loaded in this manner and their weights adjusted to give a column length of at least 20 mm. The delay is then 'cut back', to give a column length of 20 mm and then burned horizontally at atmospheric pressure. Burning times are measured using a digital timer which is started from the firing voltage and stopped using a photoelectric cell.

In order to ensure ignition, the UK delay compositions were primed with a very small amount (approx 20 mg) of SR44. Similarly the US delay compositions (with the exception of IHM-BD-11-85) were pressed with 0.4 g of the B/$BaCrO_4$ (10.3/89.7) mix. In addition, 10 mg of flash composition (SR813) was inserted into the other end of the steel delay holder to ensure that enough light output was generated on burning to stop the electronic timer. A typical delay arrangement is shown in Figure 29.

APPENDIX 10

GAP IGNITION TEST [4,5] AND 'ANGLICISED' STANDOFF TEST [1]

Gap Ignition Test

In the gap ignition test, donor and acceptor samples are separated horizontally by an air gap which is varied according to the Bruceton staircase technique. The separation distance corresponding to 50% probability of the donor charge igniting the acceptor is determined, and used as a measure of either the ignitability of the acceptor or the igniting power of the donor. About 30 to 40 separate tests are used for each donor/acceptor pair.

The test arrangement is shown in Figure 30. 100 mg samples of donor or acceptor compositions are pressed into sample tubes of 5 mm internal diameter. Acceptor samples are pressed to produce a flat end face, while the donor samples have an end face with a conical indentation. The donor sample is ignited from its rear face using an electric fuzehead. Trials using a copper calorimeter gauge have shown that a conically indented donor transmits over eight times as much energy to the acceptor face as a flat faced donor. The sample tubes are held in water cooled blocks mounted on sliding support rods. Attached to the donor block is a plate with a 4mm diameter hole, through which the donor composition combustion products must pass to reach the acceptor.

The standard donor composition, used to compare the ignitability of acceptor compositions, is 30:70 boron-potassium nitrate, and this composition is also used as an acceptor when performing regular checks on the repeatability of the technique.

'Anglicised' Standoff Test

The 'Anglicised' Standoff test was conducted using a similar apparatus to that shown in Figure 25. The Standoff tube was 13 mm diameter and made of copper.

APPENDIX 11

HIGH TEMPERATURE DSC

A cross-section of the Stanton Redcroft DSC 1500 furnace and head assembly is shown in Figure 31. The sample and reference materials are contained in 6mm diameter platinum or alumina crucibles, C, 4 mm high. The crucibles are supported on a heat flux plate, P, constructed from platinum-13% rhodium mounted in a high purity alumina cup, A. The detector plates are isolated from the main plate by slots and have integral ears for positive location of the crucibles.

The cup is fitted with a removable lid, L, and is supported on a four-bore rise rod, R, fitted into the metal base unit, M. Excellent atmosphere control is achieved by passing the desired gas directly through the DSC head, utilising one channel of the four-bore rod. Typical flow rates are in the range 25 to 75 ml min^{-1}. To minimise problems that occur with samples that bubble or creep at high temperatures, a special lid is available to enable quartz crucibles, 20 mm in length or over, to be used. Good atmosphere control can still be maintained by using the second atmosphere purging system, where a gas is passed directly through the furnace chamber via inlet, I. This can also be used for the introduction of a blanket gas, while passing a reactive gas through the head.

The head is heated by means of a water-cooled furnace, F, with non-inductively wound plantinum-rhodium windings, W. The aluminia baffles, B, reduce thermal currents, allowing high sensitivity measurements to be carried out at elevated temperatures. The furnace is provided with a motorised handling system for precise and reproducible location over the measuring head. If programmed cooling is not required at the end of the run, the furnace is lifted, rotated to position it over the built-in fan and cooled automatically. Cooling from 1800 K to ambient takes approximately 35 minutes, thus maximising run throughput. A sophisticated microprocessor-based temperature programmer enables heating rates from 0.1 K min^{-1} to 50 K min^{-1} to be selected, with the additional facility of multiple programme stages and gas switching.

APPENDIX 12

US BURNING RATE TEST

The test unit used for burning rate evaluation is a hollow cylinder 26.93 mm long, 12.45 mm OD and 5.11 mm ID. This ID was used for both IHM-BD-10-85 and IHM-BD -11-85 delays. The other delays used an ID 6.61 mm. The cylinders were made of aluminium, and although aluminium is a good heat sink and so undesirable to use with marginally propagating compositions, it is a close simulation of many in-service delay cartridge configurations. The cylinders were filled with several increments of composition and pressed to 207 MPa. An increment of igniter composition was pressed on the top. The total delay length was 25.40 mm and the length of the igniter increment was 1.52 mm.

Ignition was accomplished by functioning an ICI M-100 electric matchead which, in turn, ignited the ignition mixture and the delay composition. The delay time was measured from the time of application of electric current to the flash of an output disc. The results are the average of twenty separate tests which were conducted at 294 K. A diagram of the test unit is shown in Figure 32.

APPENDIX 13

U.S. SAFETY TESTS

Explosive materials are sensitive to impact (shock), friction, and static electricity. Impact subjects a material to high pressure, shock waves, and vibration, any of which may add sufficient energy to the system to cause initiation. Heat energy is most generally involved and may be generated either within the material itself or as a result of adiabatic compression of gases trapped in voids. Sources of execessive friction may also introduce unwanted heat energy. Static electricity releases energy to the system by means of an electrical arc.

The tests most commonly used to determine the sensitivity to initiation of both explosives and ingredients are briefly described in the following paragraphs.

Drop Tests

These tests are designed to determine the ease of initiation of detonation by impact or shock applied to a liquid or solid material. To determine impact sensitivity, a 20 mg sample is placed on an anvil, and a specified weight dropped vertically on the sample. Results are reported as mean, minimum drop height necessary to product three successive initiations or as the point of 50 percent probability of initiation. The cavity drop test is a similar test applicable only to liquid explosives. The range of test results, using a 5 kg weight, is 0 to 75 mm (high sensitivity), 100 to 325 mm (medium sensitivity), and 350 to 600 mm (low sensitivity).

Friction Test

This small-scale test measures the sensitivity of materials to initiation by friction between two metal surfaces, one a sliding block and the other a stationary wheel. The sample is placed on the block, and pressure is applied to it by the wheel which is attached to a hydraulic ram. A weighted pendulum is then swung from a predetermined position (either 45 degrees or 90 degrees may be used) so that it strikes the end of the block and imparts a velocity to it. Results are reported as the maximum force which can be applied to the wheel without causing the sample to decompose. Normally, 20 consecutive negative results must be obtained. The range of test results is hydraulic ram loading of 0 to 0.24 kN, (high sensitivity), 0.33 to 2.5 kN (medium sensitivity), and 3.33 to 4.45 kN (low sensitivity).

Electrostatic Discharge Test (ESD)

The sensitivity of the material to energy input from electrostatic discharge is determined by this test. In handling explosives, flammable solvents, and finely divided powders, there is danger of ignition of the material by static electricity. The actual hazard is determined by a number of factors such as the tendency of the material to accumulate a static charge when it is poured, mixed, sieved, or otherwise agitated; the possibility of storing a charge on plant equipment, materials, or operators; and the minimum spark energy required to initiate it.

The ESD test is designed to measure this minimum energy level. In the test, a 500 volt DC source charges a variable capacitor system, and the energy is discharged into the sample which rests on a metal test plate. To effect this discharge, a steel needle is connected to the charged condenser bank. Energy ranges run from 0.001 to 12.5 joules. The result is reported as the test level; one test interval below the test level that gave a positive result; and that level at which 20 consecutive negative results were obtained. The range of test results is: 0.001 to 0.00875 joules (high sensitivity), 0.0125 to 0.875 joules (medium sensitivity), and 1.125 to 12.5 joules (low sensitivity).

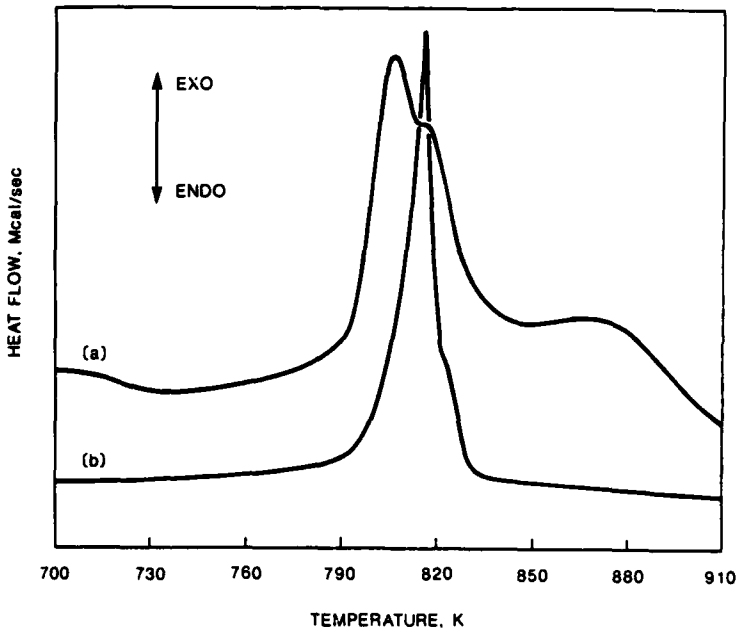


FIG. 1 DSC Curves for UK Delays (a) SR37 (b) SR38

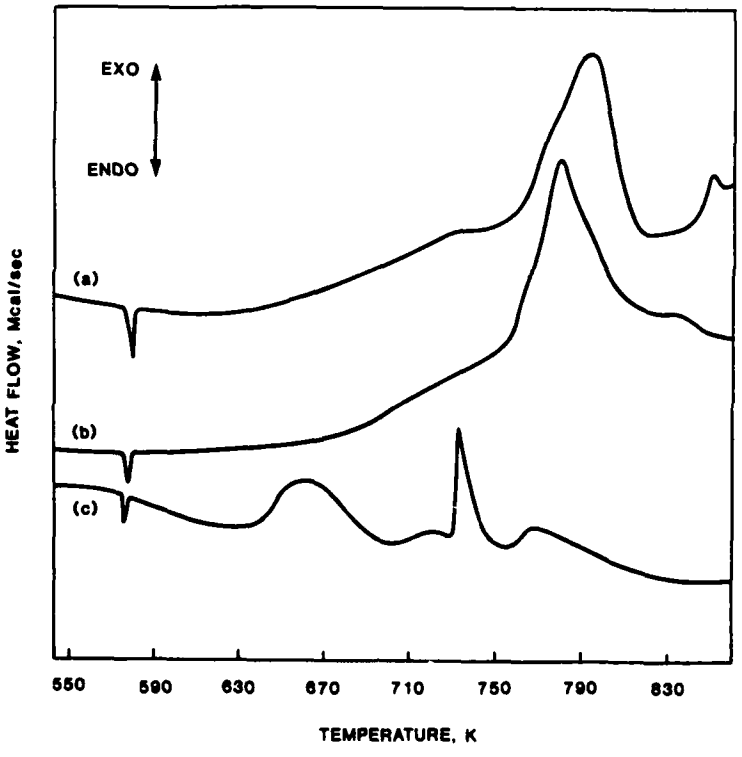


FIG. 2 DSC Curves for US Delay Compositions (a) IHM-TV-2-85 (b) IHM-WD-9-85 (c) IHM-WD-3-85

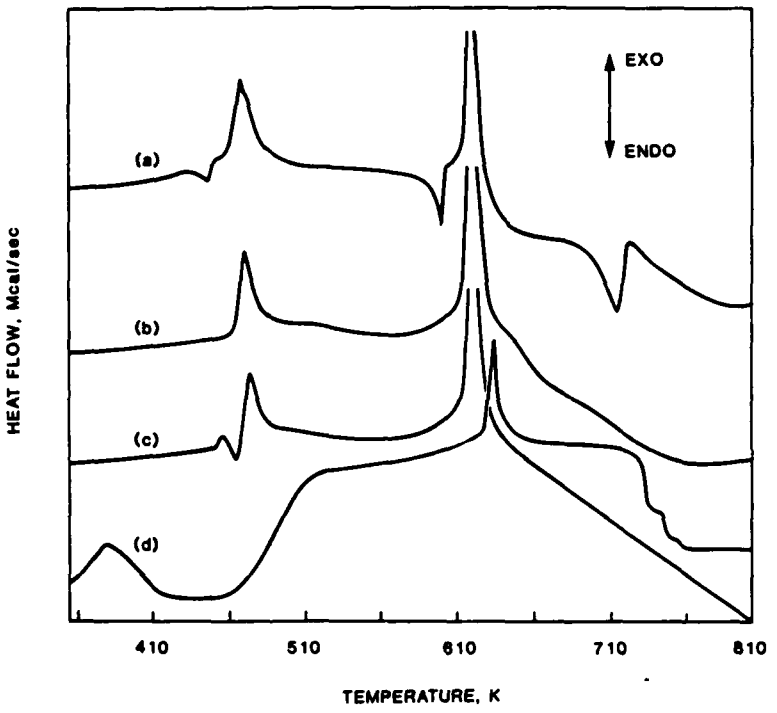


FIG. 3 DSC Curves for UK Coloured Smoke Compositions (a) PN 760 (b) PN 758M (c) PN 759 (d) PN 757

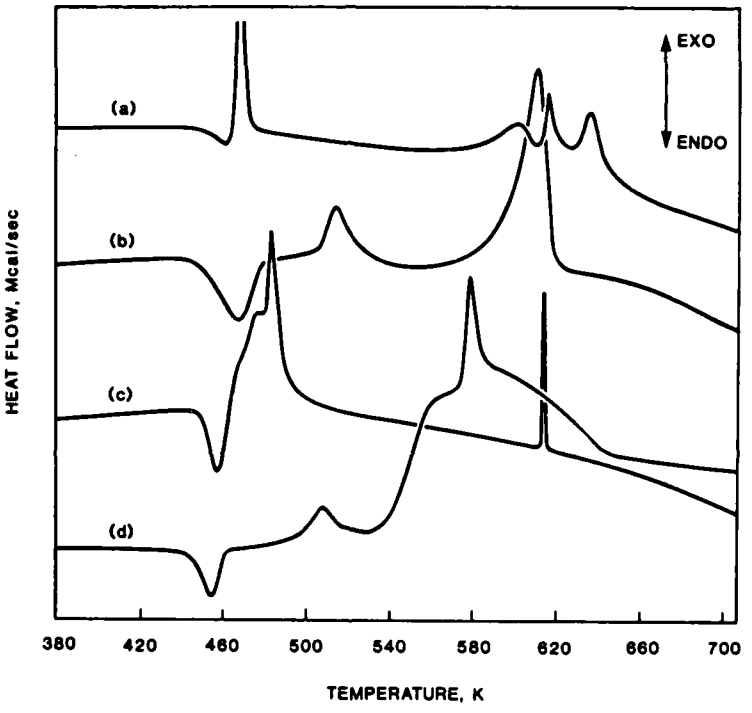
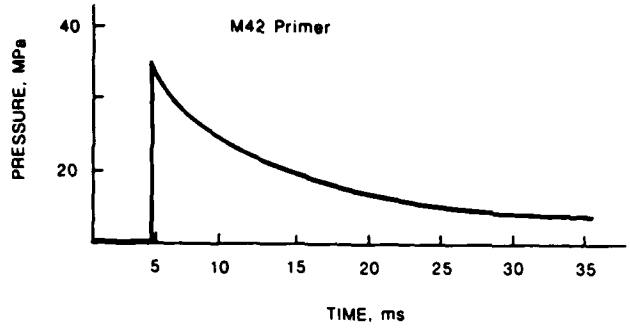


FIG. 4 DSC Curves for US Coloured Smoke Compositions (a) M-18 Green (b) M-18 Violet (c) M-18 Yellow (d) M-18 Red



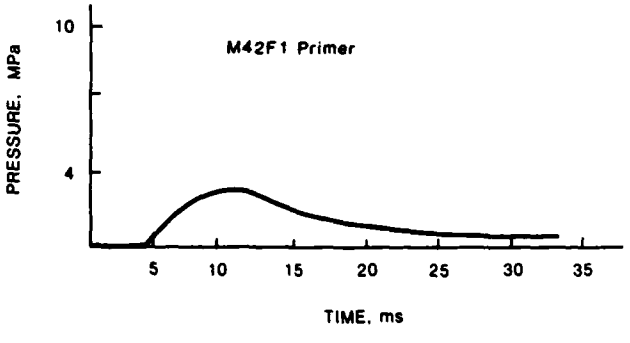


FIG. 5 Pressure Time Curves for M42 and M42F1 Percussion Primers

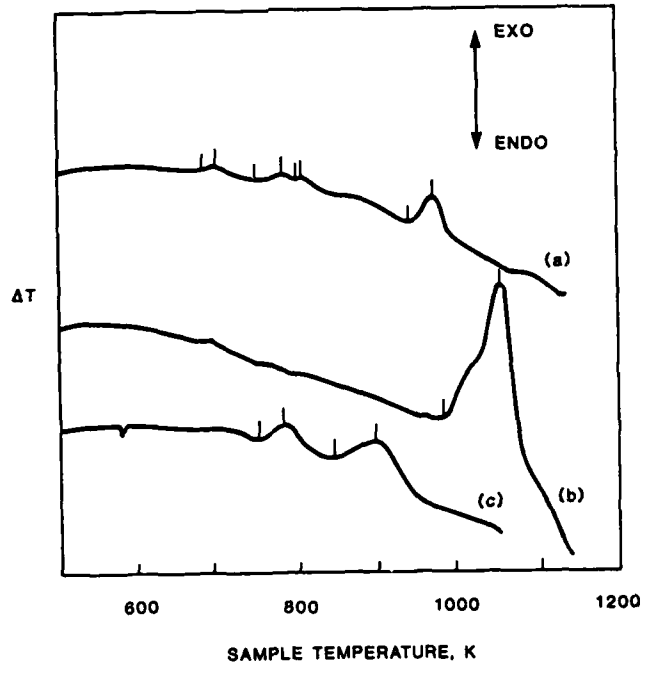


FIG. 6 DTA Curves of US Delay Compositions (a) IHM-WD-3-85 (b) IHM-BD-11-85 (c) IHM-TV-2-85

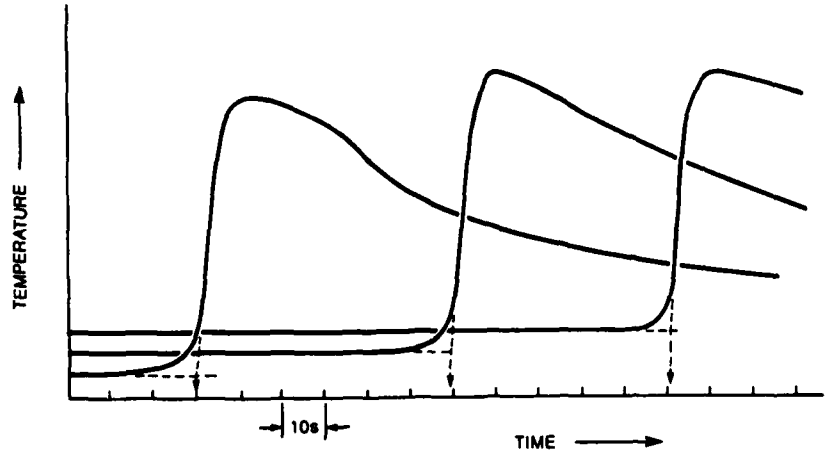
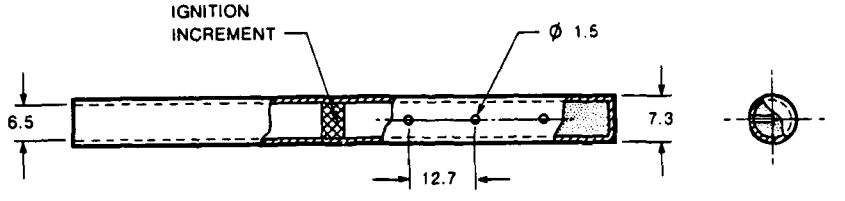
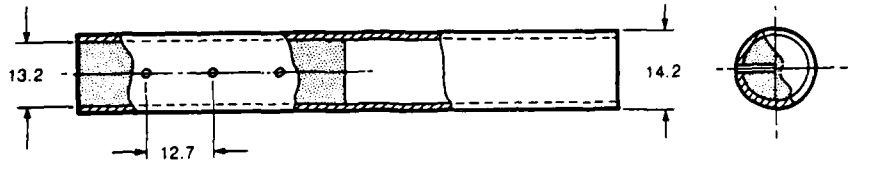


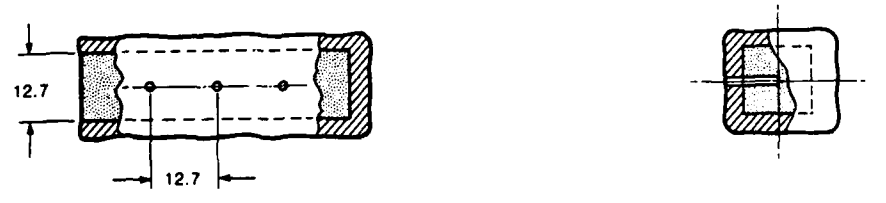
FIG. 7 Typical Thermocouple Response for Canadian Burning Rate Test



(a) Copper tube, delay compositions



(b) Phenolic tube, smoke compositions



(c) Epoxy coating, castable smoke compositions

FIG. 8 Geometries of Samples Used for Canadian Burning Rate Determination

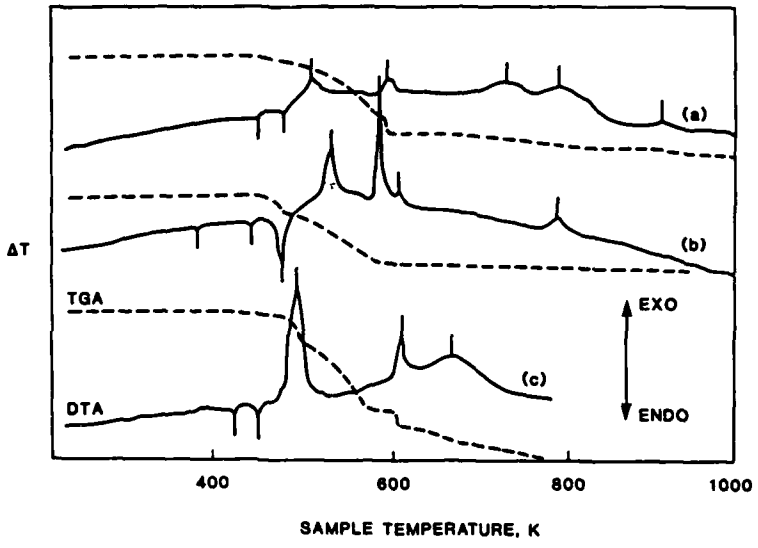


FIG. 9 DTA/TGA Curves of Red Smoke Composition (a) SK-338 (b) M-18 Red (c) PN 760

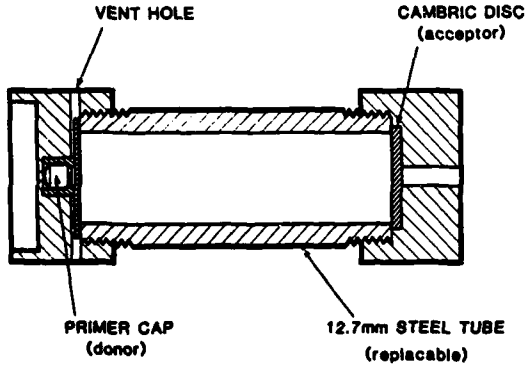
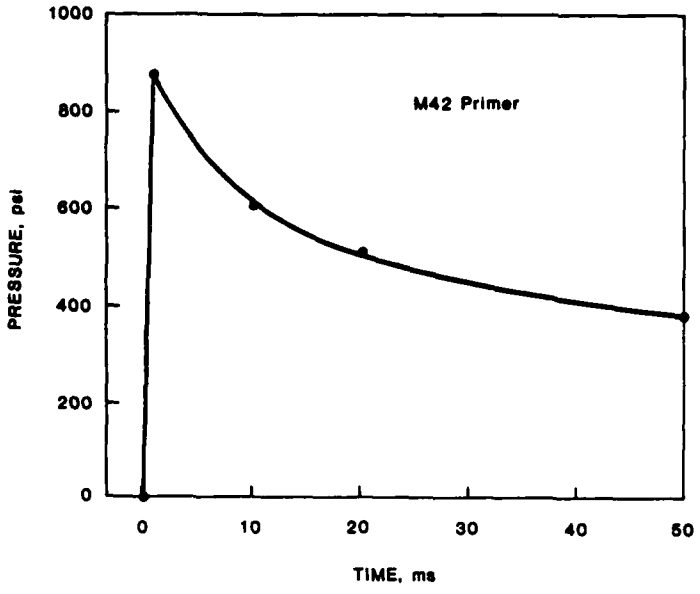


FIG. 10 Adjustable Length Canadian Flash Tube



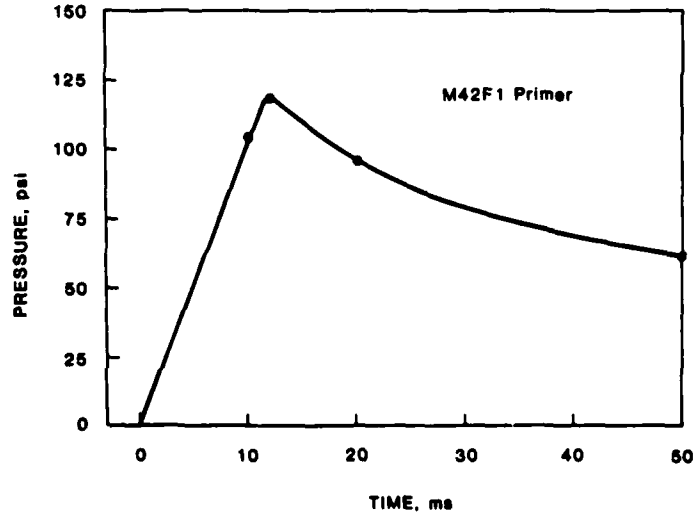


FIG. 11 Pressure Time Curves for M42 and M42F1 Percussion Primers

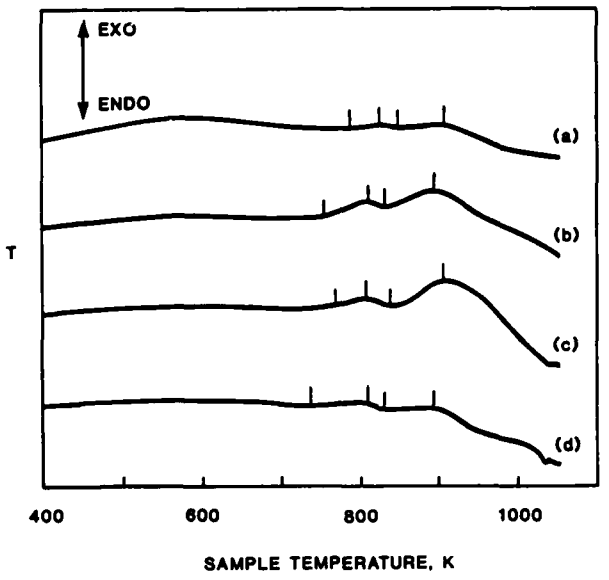


FIG. 12 DTA Curves showing Effect of Sample Mass and Heating Rate for SR37 Delay Compositions (a) 2.0 mg, 10 K min^{-1} (b) 5.1 mg, 10 K min^{-1} (c) 10.2 mg, 10 K min^{-1} (d) 5.0 mg, 5 K min^{-1}

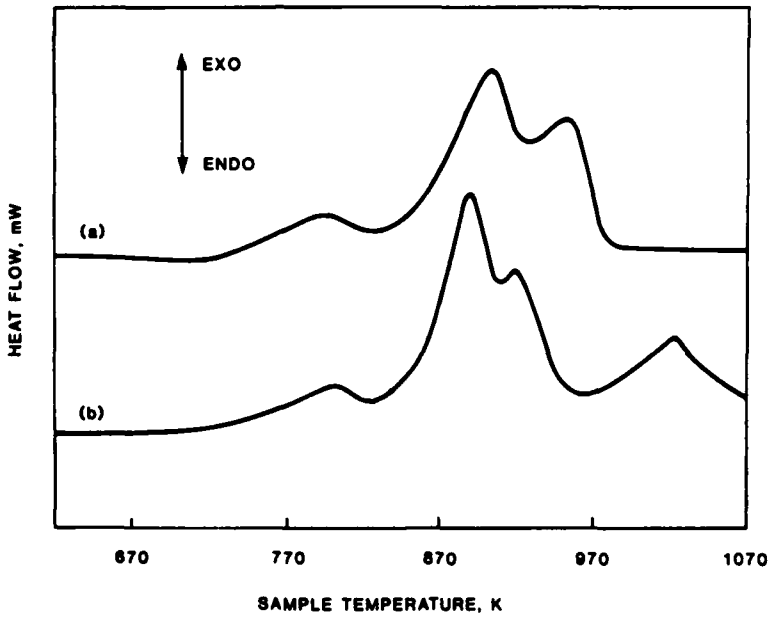


FIG. 13. DTA Curves for UK Delay Compositions (a) SR38 (b) SR37

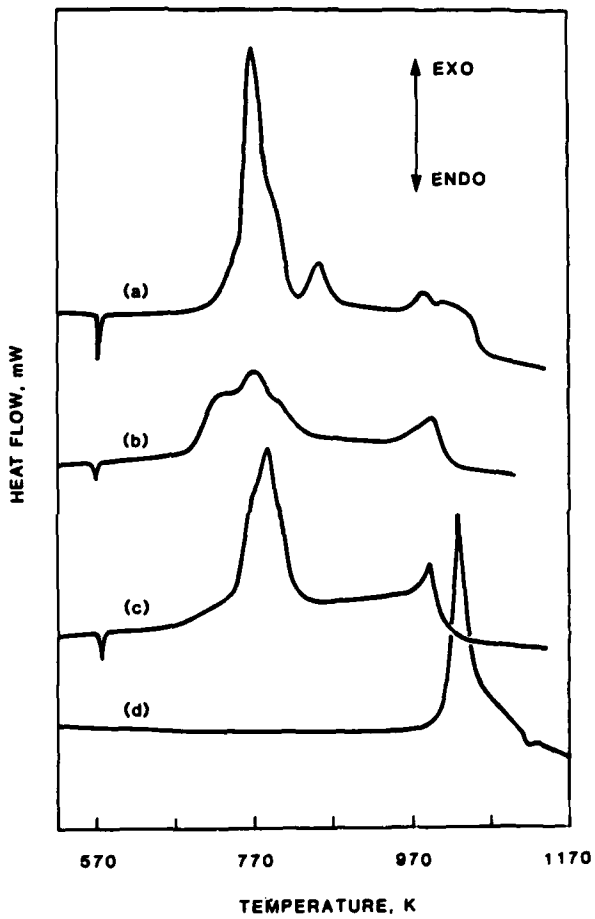


FIG. 14. DSC Curves for US Delay Compositions (a) IHM-WD-9-85 (b) IHM-WD-3-85 (c) IHM-TV-2-85 (d) IHM-BD-10-85

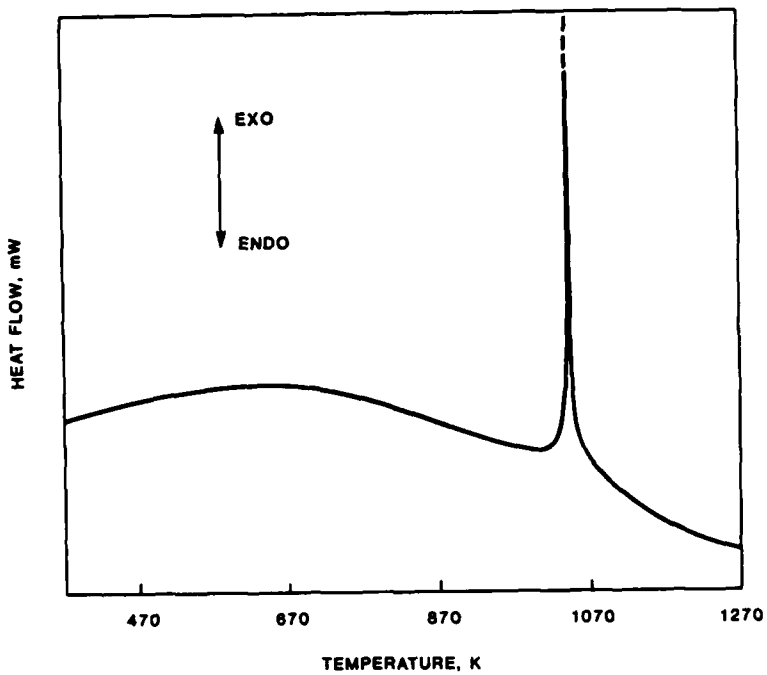


FIG. 15. Non Ignition DTA Curve for US Delay Composition IHM-BD-11-85

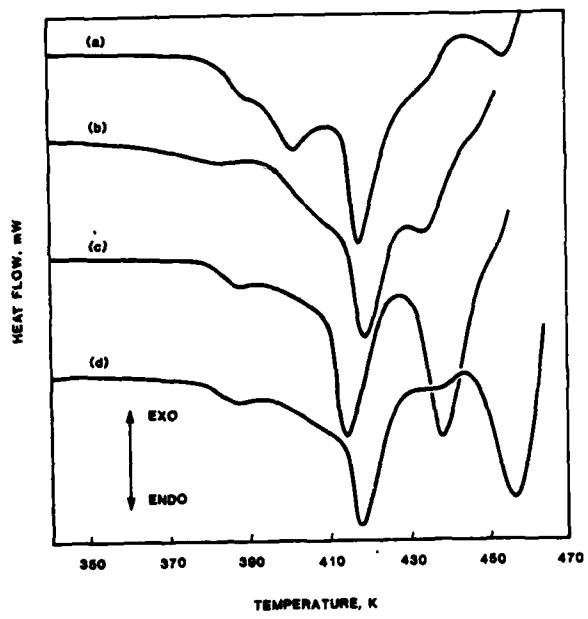


FIG. 16. DTA Curves for UK Smoke Compositions (a) PN 758M (b) PN 760 (c) PN 757 (d) PN 759

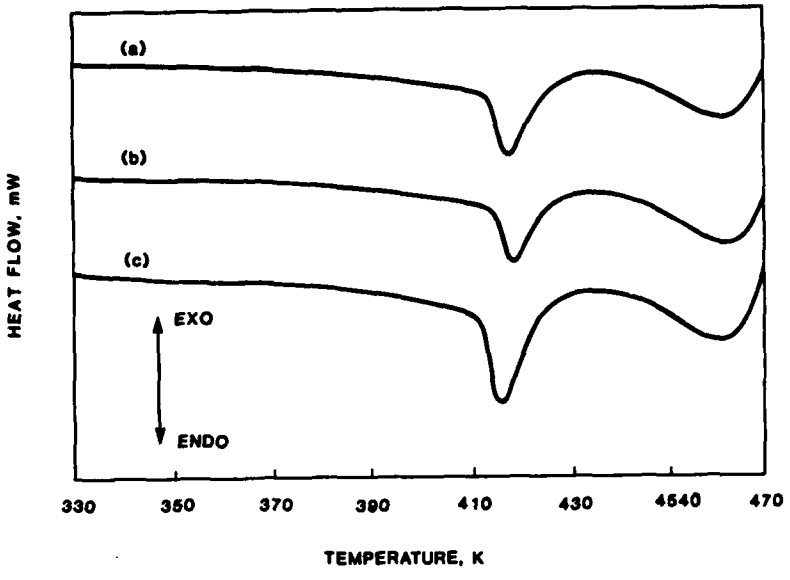


FIG. 17 DTA Curves for Canadian Compositions (a) SK356 (b) SK354 (c) SK338

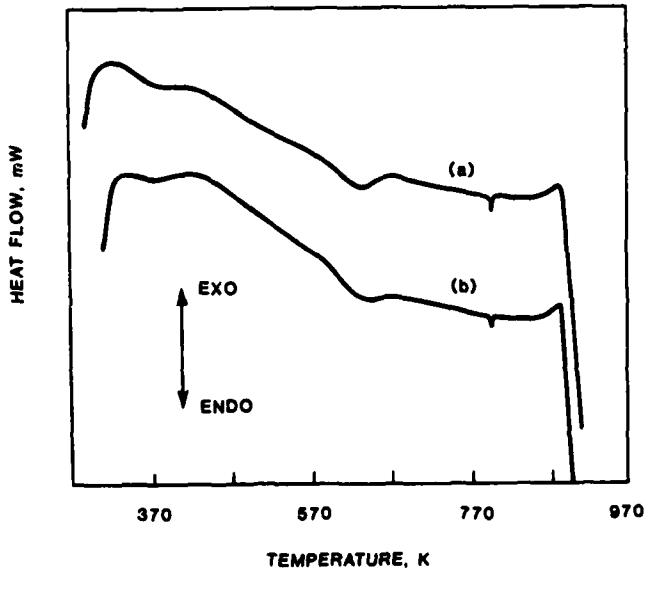


FIG. 18 DSC Curves for UK Delay Compositions (a) SR37 (b) SR38

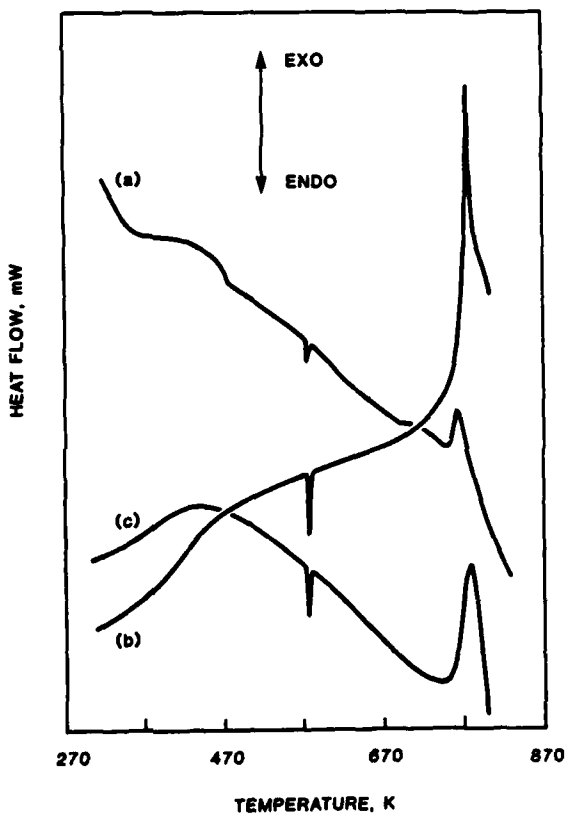


FIG. 19 DSC Curves for US Delay Compositions (a) IHM-WD-385 (b) IHM-WD-9-85 (c) IHM-TV-2-85

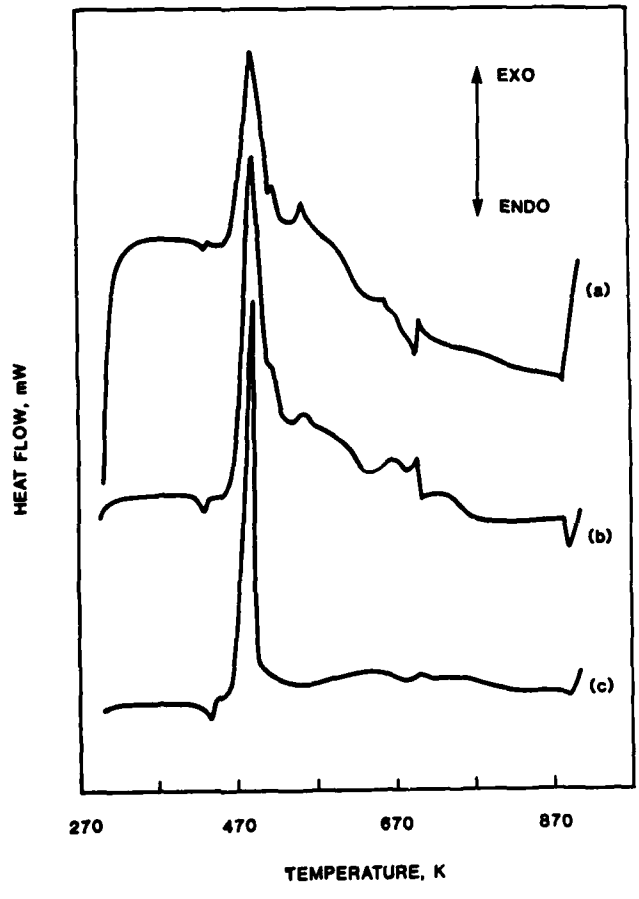


FIG. 20 DSC Curves for Canadian Smoke Compositions (a) SK 354 (b) SK 356 (c) SK 338

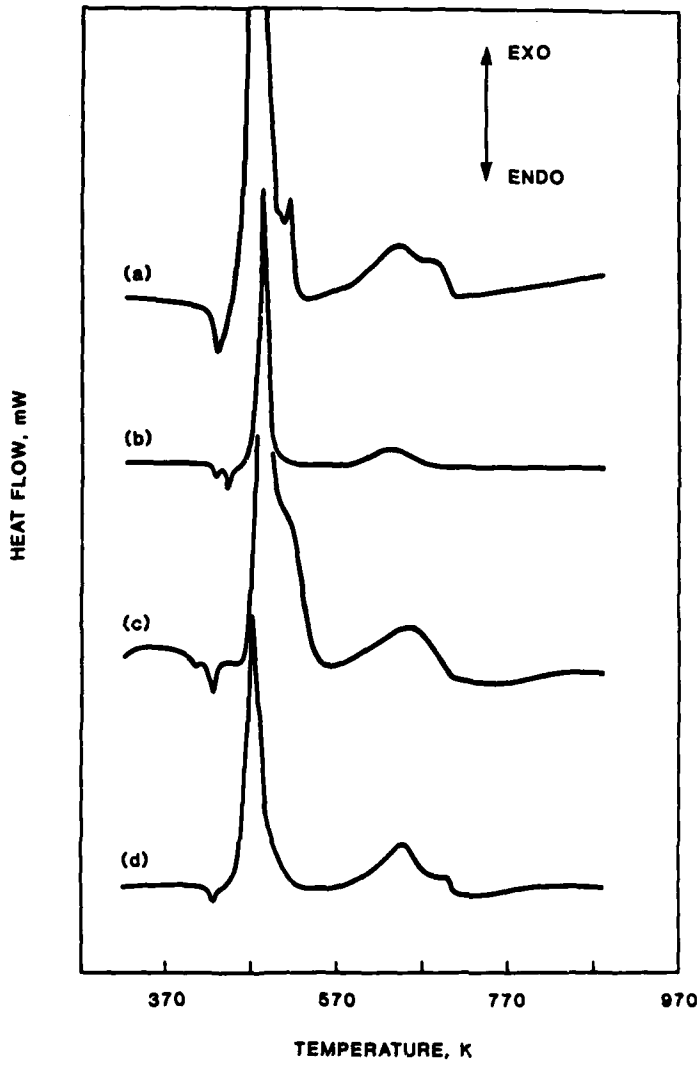


FIG. 21 DSC Curves for UK Smoke Composition (a) PN 760 (b) PN 757 (c) PN 758M (d) PN 759

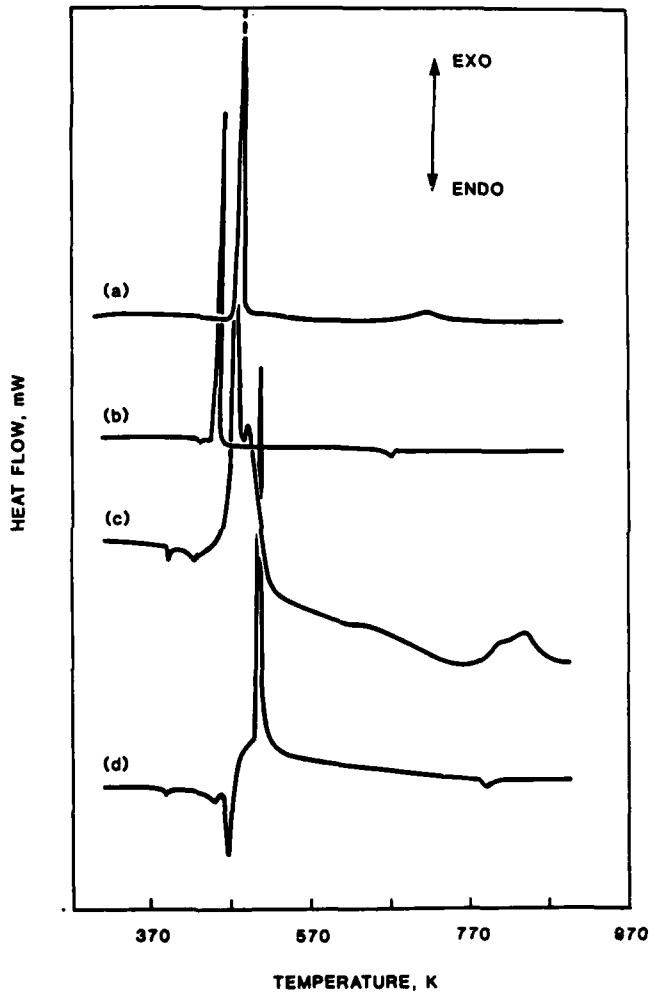


FIG. 22 DSC Curves for US Smoke Compositions (a) M-18 Green (b) M-18 Red (c) M-18 Yellow (d) M-18 Violet

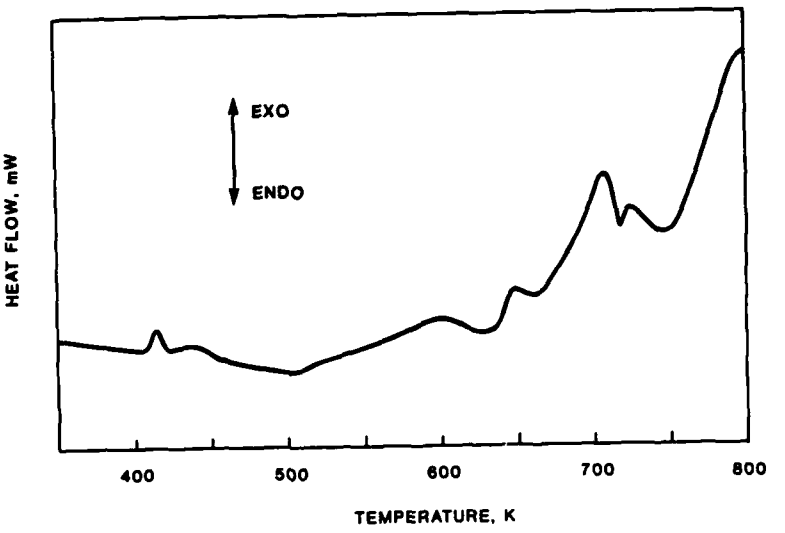


FIG. 23 DSC Curve for Australia M42F1 Primer Composition

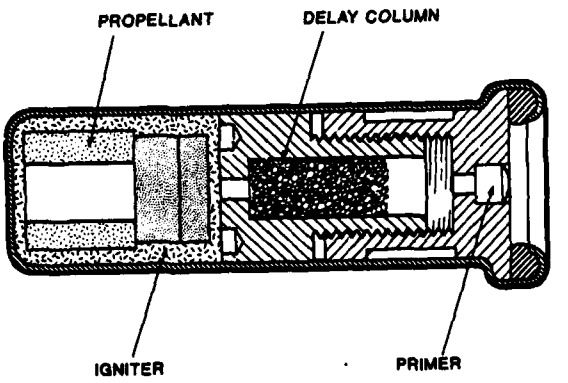


FIG. 24 US M-93 Delay Cartridges for Delay Time Measurement

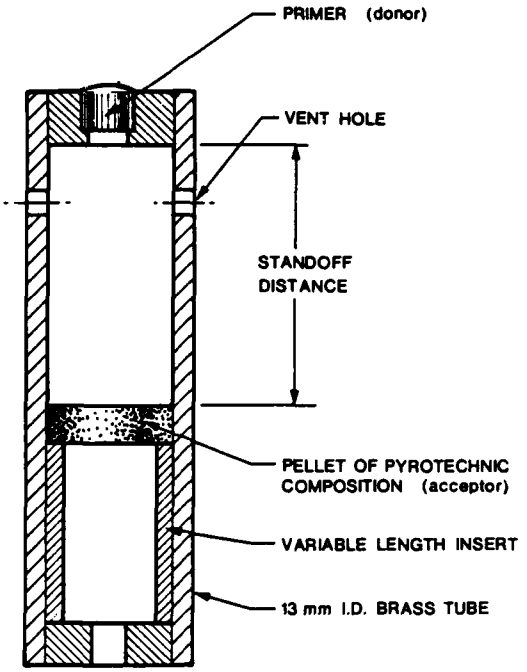


FIG. 25. Cross Section of Standoff Test Unit

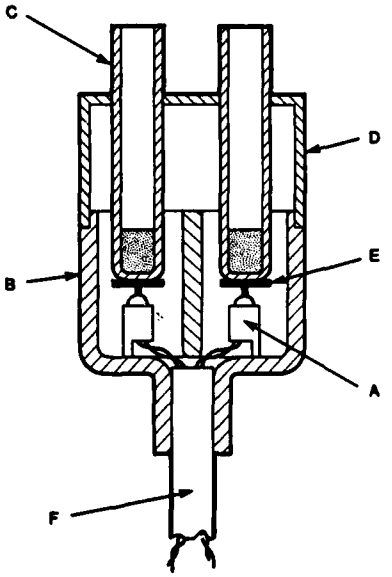


FIG. 26. Cross Section of Ignition DTA Head

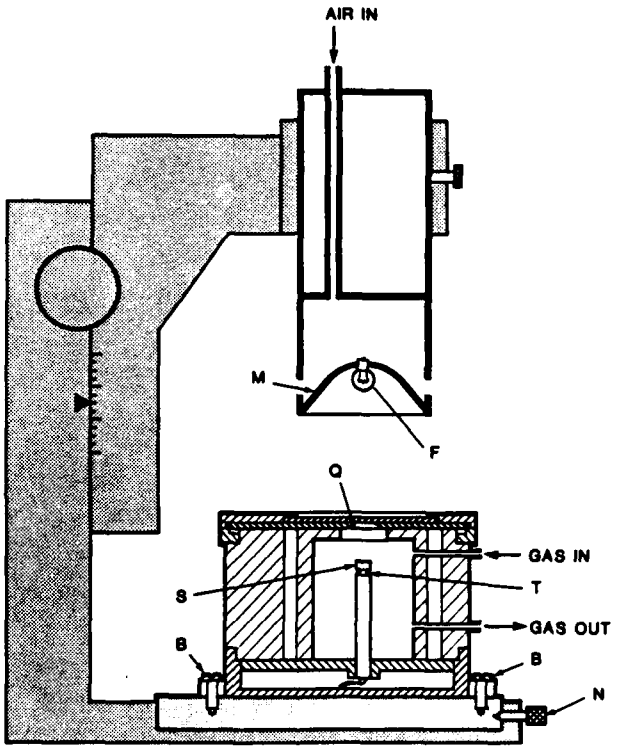


FIGURE 27. Infra-red Heating System

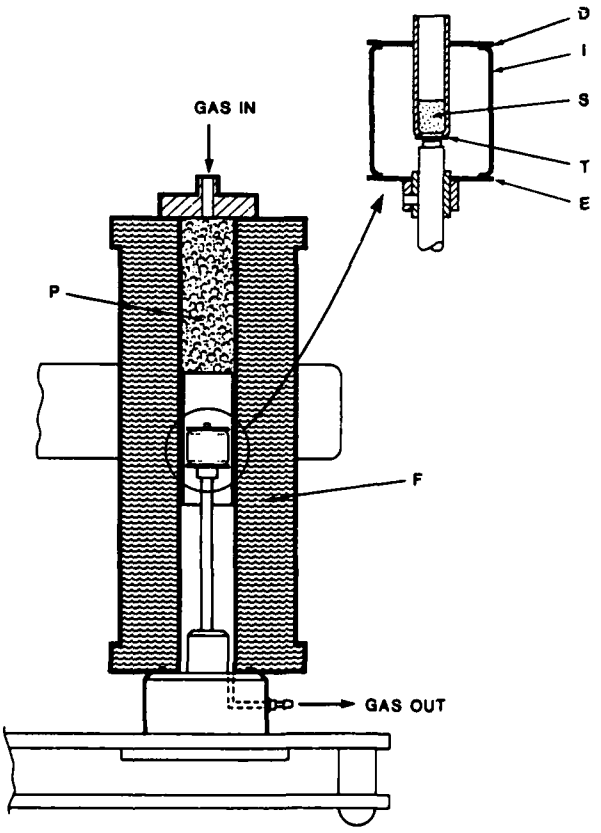


FIG. 28. Time to Ignition Apparatus

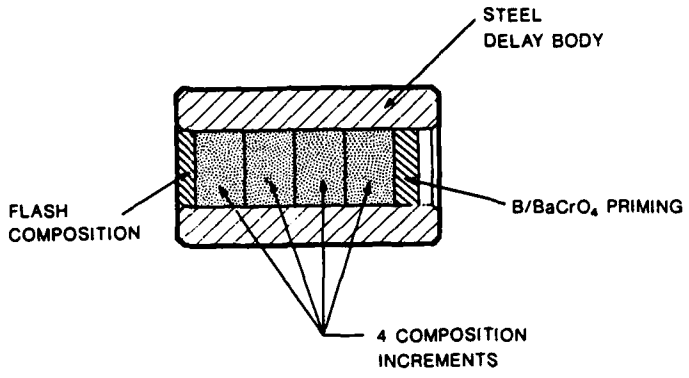


FIG. 29 Delay Burning Rate Measurement System

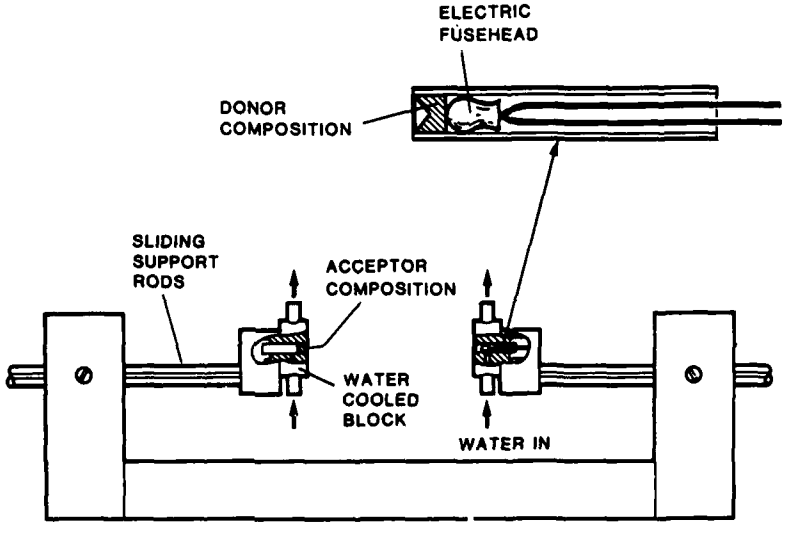


FIG. 30 Gap Ignition Test

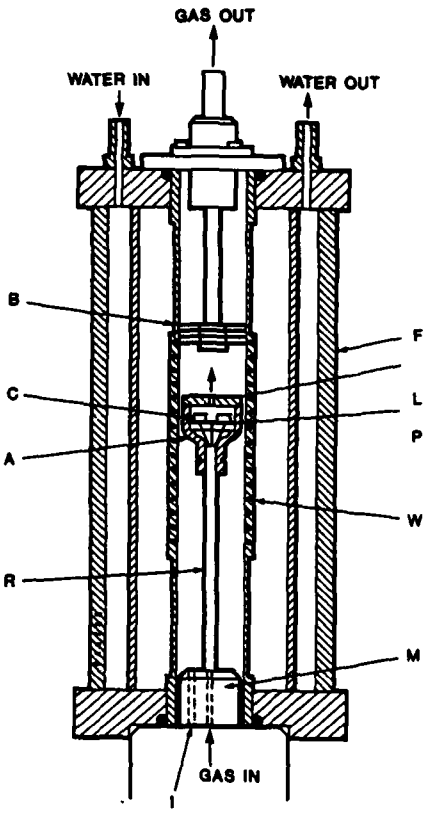


FIG. 31. High Temperature DSC Unit

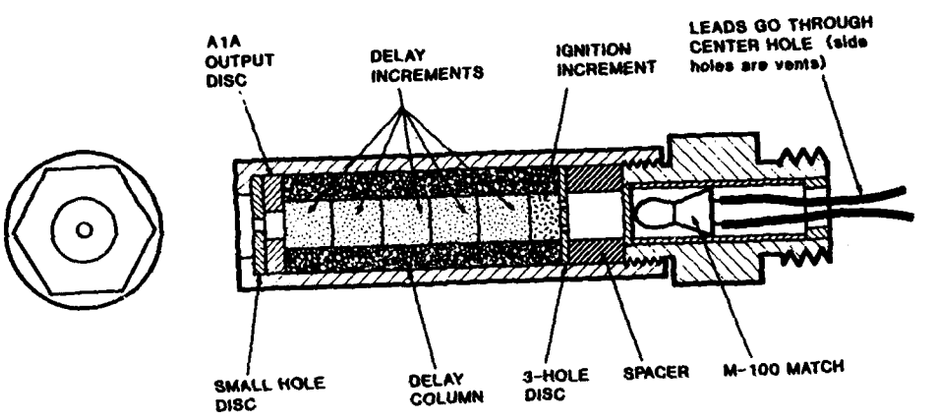


FIG. 32. US Burning Rate Test Unit